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Philadelphia, PA 19104.

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"Thermal Durability of Sapphire Optical Waveguides Processed Into High Temperature Ceramic Composites."

4. **Under the direction of (Principal Investigator):** Dr. M. A. El-Sherif

5. **Period:** April 01, 92 to June 30, 95.

6. **NASA Technical Officer:** Dr. Dan Roth

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CONTENTS

1. INTRODUCTION

Comprehensive research work was performed between the period beginning April 92 to June 95, to fabricate sapphire optical waveguides, embed them in high temperature ceramic composites and to develop a sensing technique. Commercially available plastic or silica optical fibers cannot be used at high temperatures (1000° C) since they either degrade or soften much below these temperatures. To characterize high temperature ceramics, a new kind of optical fiber and sensing technique appropriate for high temperatures need to be developed. Therefore, it was the objective of this research effort to develop an optical waveguide and sensing technique for high temperatures and embed the waveguide in ceramic composites for high temperature sensor applications. Specifically, the main objectives were:

(i) To design and fabricate sapphire optical waveguides;

(ii) To develop a sensing technique for multimode sapphire optical fibers;

(iii) To embed sapphire optical fibers in aluminum oxide (Al_2O_3) matrix; and

(iv) To study the thermal fatigue response of the processed alumina samples with embedded sapphire optical fibers.

This report discusses the results obtained in each of these areas and the conclusions that can be drawn. Significant novel concepts were proposed and considerable success obtained in this study. The important results from the study were reported in three publications:

1. M. A. El-Sherif, I. L. Kamel, F. K. Ko, M. Shaker, D. J. Roth, B. Lerch and S. Swickard, "A novel sapphire fiber optic sensor for testing advanced ceramics," Proc. of ACerS, pp. 437-444, Cocoa Beach, FL, Jan. 10-15, 1993.

2. M. A. El-Sherif, S. Hu, J. Radhakrishnan, F. K. Ko, D. J. Roth and B. Lerch, "Optical response of sapphire multimode optical sensor for ceramic composite applications," Proc. of SPIE, Vol. 2072, pp. 244-251, Boston, MA, Sept. 8-10, 1993.

3. M. A. El-Sherif, W. Shih, Z. Cai, J. Radhakrishnan, S. Hu, F. K. Ko, D. J. Roth and B. Lerch, "Development of a fiber optic sensor for ceramic materials characterization," Proc. of ACerS, pp. 373-381, Cocoa Beach, FL, Jan. 9-14, 1994.

These publications appear as appendix A in this report.

2. DESIGN AND FABRICATION OF SAPPHIRE OPTICAL WAVEGUIDES

Optical grade sapphire fibers are grown as high quality single crystal fibers. Unlike commercially available silica or polymer optical fibers, sapphire fibers are uncladded. A cladding layer is required in optical fibers to confine propagating optical signals within the fiber core and limit attenuation and losses. Therefore, to fabricate sapphire optical waveguides for sensor applications, a cladding layer needs to be developed. In the first six months of this research study an excellent cladding layer consisting of polycrystalline aluminum oxide was developed.

The developed technique was based on using a polymerizable monomer as the carrier for very fine alumina particles. A solution of acrylic acid in water with benzoyl peroxide (BPO) as the initiator and N,N-dimethyl para toluidine (DMPT) as the accelerator was used to dissolve alumina particles and form a suspension. The sapphire fiber to be coated was immersed in the suspension; the polymerization reaction allowed to proceed. The BPO in the solution formed free radicals which reacted with acrylic acid monomer. Before the end of complete polymerization, the fiber was pulled out of the suspension. The coated fibers

were left to dry at room temperature for 24 hours, and then heated for evaporation of the polymer. This was followed by sintering of the fiber coating to obtain a uniform cladding of polycrystalline alumina.

The concentration of alumina, acrylic acid, BPO and DMPT and the length of time for which the polymerization reaction was allowed to proceed are important parameters that determine the uniformity and thickness of the cladding. These parameters depend on the diameter of the fiber that is required to be coated. By successfully adjusting these conditions, a uniform coating with a good interface between the core and the cladding was achieved. An exhaustive account of this work was provided in the progress report submitted on October 15, 1992. A copy of the progress report is attached with this report as appendix B.

3. A SENSING TECHNIQUE FOR SAPPHIRE OPTICAL FIBERS

Sapphire fibers possess reasonably good optical properties, but are difficult to be fabricated into single mode fibers. Most conventional high sensitive techniques are either based on phase modulation or polarization modulation and, therefore, require single mode fibers for propagation of coherent light. Since available sapphire fibers are multimode fibers, as opposed to single mode, these techniques are not suitable. For multimode fibers, an inexpensive and reasonably sensitive technique was developed recently by the PI of this project, Dr. M.A. El-Sherif.

The principle of operation of the sensing technique is based on spatial modulation of the modal power in multimode fibers. Within a multimode optical fiber, optical signals propagate according to the modal structure of the fiber and the boundary conditions. Altering the boundary conditions of an optical fiber due to an external perturbation induces

modal coupling and results in modal power distribution modulation. This energy exchange or coupling is referred to as spatial intensity modulation since it results in spatial variation of the light pattern within the fiber. Comparative measurements of the distribution and subsequent redistribution of the modal power can be accomplished by scanning the far field pattern at the fiber end using a CCD camera or array of photodetectors. This technique can be employed as a sensitive mechanism for measuring perturbations induced by external mechanical stresses. The sensitivity of this type of fiber optic sensor is related to the modal structure of the fiber and to the medium surrounding it. The launching conditions of the light determines the initial modal power distribution (MPD) and is the most critical factor for sensitivity. In order to improve the sensitivity of the device, it is preferred to excite higher order modes within the fiber. This is done by launching an optical beam slightly off-axis to the fiber. In this way, the far field pattern becomes totally dark at the center; any induced power coupling from higher to lower order modes i.e., from the periphery to the center, can be detected easily. The quality of the fiber end faces and the fiber coating are additional factors that influence the level of sensitivity.

The spatial intensity modulation technique was successfully to sapphire optical fibers inspite of the crystalline nature of sapphire. The annual report submitted on July 10, 1993 and attached here as appendix C provides details on the experimental set up and results obtained using this technique.

4. PROCESSING OF ALUMINA MATRICES WITH EMBEDDED SAPPHIRE OPTICAL FIBERS

Several approaches were advocated in the progress and annual report to process alumina matrices with embedded sapphire optical fibers. These approaches included slip casting using a plaster of paris or gypsum mold and pressure filtration. Due to the superior nature

of results obtained using the pressure filtration method, this method was adopted. The pressure filtration process consisted of the following steps: A slurry was prepared by mixing alumina powder with a binder and solvent. Then, it was cast under pressure in a special mold (to accomodate and protect optical fiber leads). The pressurized casting led to filtration of the solvent and at the same time a compaction of the powder. The fiber was embedded during the casting process and after casting, the green body was taken out of the mold and let to dry. Finally, the dried green body was heated to remove the binder and sinter the alumina matrix. Preliminary work with illustrations of the pressure filtration process was reported in the annual report submitted on July 10, 1993. The following sections provide exact steps involved in the process and complement the description in the annual report.

4.1 Slurry Preparation

Sample preparation began with alumina in powder form. About 17 g of alpha alumina powder (type AKP - 15, 99.99% pure and average size of 0.7 μm, supplied by Sumitomo Chemical Co, New York, NY) was mixed with distilled water and 2 cc of binder solvent (Rhoplex emulsion, supplied by Rohm and Haas, Philadelphia, PA) was added. The total volume was made up to 28 cc by adding more distilled water. The above measure of alumina powder led to a sintered ceramic coupon of approximate dimensions 40 x 20 x 5 mm. The use of ultrapure alpha alumina (99.99%) eliminated the deleterious effects of impurities like calcium oxide and silicon dioxide during sintering.

The slurry was stirred well using a magnetic stirrer in order to ensure homogeneity and an ultrasonic processor was used to break down any powder agglomerates. The pH was adjusted to about 4 before the addition of binder, so that the particles developed a positive

surface charge. This was done to repel particles from one another and thus prevent flocculation.

3.2 Pressurized Casting

The mold used to make the green body was cuboidal in shape, made of 4 plexiglass plates on the sides, one plexiglass plate on top, and a porous stainless steel plate at the bottom to facilitate draining of water. First, the bottom porous stainless steel plate was surrounded by the four side plates of the mold and held in place with the help of 3 C-clamps. About 50% of the slurry was poured into the mold and a plexiglass plate was placed on top. Then a compressive load was applied on the top plate at the rate of 30-40 lb a minute. The load was increased to a maximum of 400-500 lb (this corresponded to a pressure of 200-250 psia, since the dimensions of the sample pressed was 2 x 1 x 0.1 in), and maintained at the final load for 10 minutes. The load was removed and the optical fiber was placed on the pressed body by inserting it through the pre drilled holes on the side plates of the mold. Finally, the rest of the slurry was poured and the procedure was repeated again by applying a compressive load. However, this time the maximum load was increased to about 900-1500 lb (450-750 psia). The pressed green body was taken out of the mold by removing the side plates of the mold first, so that the leads of the optical fiber were left intact. The green body was placed on a porous base to dry for about 48 hours.

3.3 Sintering

The sample was placed in a furnace and the temperature increased at a rate of 1° C per minute until a temperature of 600° C was reached. Debinding was allowed to occur at 600° C for one hour; then the temperature was increased to 1400° C for sintering. The sample was left at 1400° C for 4 hours and then cooled to room temperature at the same rate.

3.4 Discussion

The main parameters involved in this process are the viscosity of the slurry mixture, the loading rate during pressing, the maximum compressive load, the drying of the green body and the thermal cycle during sintering. Viscosity plays a significant role in the flow behavior of the slurry while pressing. Low viscosity can lead to the powder being transported with the liquid during pressing, whereas a high viscosity restricts the flow and may lead to formation of voids. Viscosity is determined by the amount of water and binder used. A correct amount of binder can improve the plasticity, reduce the liquid migration rate and improve the green strength. The selected emulsion in this process was good for admixing at room temperature, soluble in water and greatly increased the strength of the green body. The ratio of the ingredients used in the process was determined such that the relative density and strength of the green body was maximized.

Pressing and drying procedures are critical to ensure uniformity in the density of the ceramic final product. A slow loading rate is essential to allow sufficient time for the slurry to flow and lead to good filling to avoid pressure gradients, density variations and void formation. Sudden decompression or unloading should also be avoided, so as to minimize elastic expansion or springback. The drying of the green body must be carefully controlled to avoid differential shrinkage, which can lead to warping and defects such as cracks in the product. Preliminary drying by natural convection on all faces followed by furnace drying at a very slow rate was found to minimize differential shrinkage. During the firing process, sufficient time was allowed for binder burnout and a slow rate of heating and cooling was used to avoid excessive thermal stresses. A stress buildup can give rise to warping or even

crack formation. The developed sintering schedule resulted in samples with no cracks or bending.

Several alumina coupons with embedded optical fibers were successfully fabricated, after addressing all the above issues. To test the durability of the prepared samples, thermal fatigue response tests were performed on them.

4. THERMAL FATIGUE RESPONSE TESTS

To check the thermal fatigue resistance of the processed samples, heat treatments were performed on 10 ceramic samples with embedded optical fibers. Table 1 shows the temperatures at which heat treatments were performed, the holding times at these temperatures and the number of cycles. For example, sample # 1 was heated to 900° C for an hour and cooled to room temperature, whereas, sample # 9 was heated to 1300° C, held for a minute and cooled to room temperature during a single cycle and the cycle repeated 10 times. In addition to varying the temperature, holding time and number of cycles, two different heating and cooling rates were used. Samples treated at 900° C were subjected to a heating rate of 20° C/min and those at 1300° C were subjected to a heating rate of 40° C/min. The cooling rates were approximately the same as the heating rates. From the table it is seen that the objective of the heat treatments on samples 1, 2, 3, 6, 7 and 8, was to look at the behavior of the samples when exposed to high temperatures for extended periods, whereas, heat treatments on samples 4, 5, 9 and 10 were performed to look at thermal cycling resistance.

After heat treatment, each sample was carefully examined by naked eye, optical microscope with up to 400x magnification, and a scanning electron microscope (SEM) in order to examine the fiber/matrix interface between the fiber and matrix. Cracking in the ceramic

matrix and debonding between the fiber and matrix are the two major defects that can arise due to thermal cycling. Presence of either of these defects in the sample can result in faulty detection of stress levels in the sample when the optical fiber is used as a stress sensor or can even render the sensor completely useless. The observations performed by both simple naked eye examination and by optical microscope revealed no signs of microcracking in the samples. In addition to this, samples 8 and 10 were cut in the transverse direction and the cross sections examined using an SEM. Figure 1 shows the micrograph of a transverse section of sample 10. The interface between the fiber and matrix is seen to be perfect, without any debonding. This result is expected, since both the matrix and fiber are alumina and fusing together during the sintering process. Hence, the samples are impervious to both thermal cycling and exposure to high temperatures for extended periods.

5. CONCLUSIONS

The following important conclusions can be drawn form this research study:

(i) A novel chemical deposition process was developed to coat sapphire optical fibers with polycrystalline alumina cladding.

(ii) Appropriate process parameters were developed to obtain claddings of good quality with thickness in excess of 20 μm.

(iii) A pressure filtration followed by sintering procedure was developed to embed sapphire optical fibers in alumina composites.

(iv) Thermal fatigue response tests performed on samples of alumina with embedded sapphire optical fibers confirmed the thermal durability and resistance of the process samples.

Table 1 Heat treatments performed on alumina samples with embedded sapphire fibers.

Sample #	Temperature (in °C)	Holding Time	# of cycles
1	900	1 hour	1
2	900	24 hours	1
3	900	100 hours	1
4	900	1 min	10
5	900	1 min	100
6	1300	1 hour	1
7	1300	24 hours	1
8	1300	100 hours	1
9	1300	1 min	10
10	1300	1 min	20

Figure 1 Transverse section of sample 10 after thermal fatigure testing.

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DEVELOPMENT OF A FIBER OPTIC SENSOR FOR CERAMIC MATERIALS CHARACTERIZATION

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ABSTRACT

A novel sapphire optical waveguide has been developed for high temperature applications. A single crystal optical grade sapphire fiber was coated with two layers of alumina and SiC in order to construct an optical waveguide. Opto-mechanical tests were performed in order to examine the fiber response to external stresses. In this paper, the optical response of sapphire fibers to microbending is briefly presented and the development of a processing methodology to embed these optical fibers in alumina matrix is reported. Several coupons were successfully fabricated and examined by thermal cycling tests.

INTRODUCTION

The development of embedded sapphire optical waveguides offers significant potential for in-situ monitoring of ceramic materials under operating conditions. These embedded sapphire fiber-optic sensors can be used for real-time evaluation of materials damage and stress distribution in a high temperature environment (over 1000° C). Unlike conventional silica fibers, it has been reported that sapphire fiber sensors are operable up to 1600° C [1].

The principle of operation of the developed optical sensor is based on spatial modulation (SM) of optical signals propagating through a multimode optical fiber (i.e. modal power distribution modulation) due to external perturbations. Application of the SM technique to multimode silica fibers has been reported earlier [2-6]. Studies show that the developed SM technique is inexpensive and highly sensitive to external perturbations. Recently, the spatial modulation technique was applied to sapphire fibers and shown to be feasible, in spite of the crystalline structure of sapphire, which would result in interference phenomena (unlike the amorphous nature of silica). The response

of the sapphire fiber to lateral compression loads has been presented [7]. We include here a brief description of sensor operating principles and present results of preliminary micro-bending tests performed on sapphire fiber.
Sapphire optical fibers are single crystal unclad alumina fibers (in contrast to conventional silica fibers which have a cladding layer). It is imperative to coat the sapphire fiber with a cladding layer in order to confine the optical signal within the fiber core and limit attenuation and radiation losses. A deposition technique based on a polymerization reaction to coat sapphire fibers with a cladding layer of polycrystalline alumina, has been reported earlier [8]. In addition, it is advisable to have an external coating to protect the embedded fiber from its surrounding environment. For this layer of coating, silicon carbide (SiC) is preferred to other materials due to its mechanical and thermal properties.

In addition to presenting the results of preliminary micro-bending tests this paper reports a processing technique for embedding the coated fiber in an alumina matrix. This would be a significant step in terms of the actual application of sapphire fibers as a intrinsic sensors, instead of the normal extrinsic sensor applications reported thus far [9,10]. Currently, there is no quick and easy way to produce a ceramic matrix having an embedded optical fiber with exposed leads. The key issues in embedding optical fibers in ceramic matrices are to avoid any defects such as cracks or debonding between the fiber and the matrix and protecting the leads of the fiber during the impregnation process. The current approach taken to embed optical fibers is based on a pressure filtration method [11]. A slurry is prepared by mixing the ceramic powder with a binder and solvent. Then, it is cast under pressure in a special mold. The pressurized casting leads to filtration of the solvent and at the same time the compaction of the powder. The fiber is embedded during the casting process and after casting, the green body is taken out of the mold and let to dry. The dried green body is then heated for binder removal and sintering the alumina matrix.

In situations where the material is subjected to high temperature cycling rather than being maintained at a constant temperature, it is important to examine the processed samples by thermal cycling. Therefore, thermal cycling at different temperatures for different periods of time were performed to check the fatigue response of the matrix with an embedded sapphire fiber.

OPTO-MECHANICAL TESTING OF SAPPHIRE FIBERS

The principle of operation of the optical sensor is based on SM of optical signals propagating through the fiber due to external perturbations. The details of the experimental setup and the modal coupling that gives rise to spatial modulation was reported earlier [7]. Opto-mechanical testing was performed by applying lateral compression loads to the fiber and measuring induced changes in the optical far-field pattern at the fiber output. For the

present work, the experimental setup was slightly modified to include a microbend transducer that acts as a modal perturber. Figure 1 shows the block diagram of the modified experimental setup. The 5-point bending transducer consists of two metal plates. The lower plate is stationary and has three equally spaced sharp ridges 0.9 cm apart, while the upper plate is mobile and has two ridges 0.9 cm apart. The optical fiber is placed on the lower plate and the upper plate is used to apply quantified sinusoidal displacement to the fiber. The key issues in the operation of the sensor are briefly described here. For a detailed explanation of the spatial modulation technique, the reader is referred to previous work [3,4,7]. Light is launched into the optical fiber at a special angle (about 8° off axis), so that the higher order modes are selectively excited. The result is a far-field pattern that has a bright ring with the center totally dark as shown is figure 2. With application of a sinusoidal displacement, light from the higher order modes is coupled to the lower order modes, resulting in an increase in intensity near to the center. A gradual increase in intensity at the center of the far-field pattern can be detected, as the sinusoidal displacement is increased slowly. To illustrate the phenomenon, the far-field pattern at one particular displacement (7 mm approximately) is shown in figure 3. For more bending, more power is transferred to the lower order modes. As the amplitude of the sinusoidal undulation increases, much more power is shifted to the center of the pattern for bending amplitude about 10.5 mm, as shown in figure 4. A simple plot of the normalized optical intensity against vertical displacement can be obtained easily. The exact relationship between the normalized intensity at the center and displacement is yet to be derived. A simple approach for practical application in future would be to replace the CCD camera with a single photodetector positioned on the fiber axis to collect the power in the lower order modes. The measured power can be related to the displacement experienced by the fiber.

PROCESSING OF ALUMINA MATRIX WITH EMBEDDED OPTICAL FIBER

In the following sections, the focus will be on the processing technique developed for embedding optical fibers in alumina matrix.

Slurry Preparation

Sample preparation starts with alumina in a powder form. About 17 g of alpha alumina powder (type AKP - 15, 99.99% pure and average size of 0.7 μm, supplied by Sumitomo Chemical Co, New York, NY) is mixed with distilled water and 2 cc of binder solvent (Rhoplex emulsion, supplied by Rohm and Haas, Philadelphia, PA) is added. The total volume is made up to 28 cc by adding more distilled water. The above measure of alumina powder leads to a sintered ceramic coupon of approximate dimensions 40 x 20 x 5 mm. The use of an ultrapure alpha alumina (99.99%) reduces some of the

deleterious effects of impurities like calcium oxide and silicon dioxide during sintering [12].

The slurry is stirred well using a magnetic stirrer to ensure homogeneity and an ultrasonic processor is used to break down powder agglomerates. It is also advisable to adjust the pH value to about 4, before the addition of binder, so that the particles develop a positive surface charge. This electrostatic charge repels particles from each other and prevents flocculation.

Pressurized Casting

The mold used to make the green body is cuboidal in shape, made of plexiglass and contains a porous stainless steel plate at the bottom to facilitate the draining of water. The side plates of the mold are held in place with the help of 3 C-clamps. About 50% of the slurry is poured into the mold and a plate is placed on top. Then a compressive load is applied on the top plate at the rate of 30-40 lb a minute. The load is increased to a maximum of 400-500 lb (this corresponds to a pressure of 200-250 psia, since the dimensions of the sample pressed is 2 x 1 in), and maintained at the final load for 10 minutes. The load is removed and the optical fiber is placed on the pressed body by inserting it through the pre drilled holes on the side plates of the mold. The rest of the slurry is poured and the procedure is repeated again by applying a compressive load. However, this time the maximum load is increased to about 900-1500 lb (450-750 psia). The pressed green body is taken out of the mold by removing the side plates of the mold first, so that the leads of the optical fiber are left intact. The green body is placed on a porous base to dry for about 48 hours.

Sintering

The sample is placed in a furnace and the temperature increased at a rate of 1° C per minute until 600° C. Debinding is allowed to occur at 600° C for one hour and the temperature increased to 1400° C for sintering. The sample is left at 1400° C for 4 hours and then cooled to room temperature at the same rate. The heating and cooling rates play an important role, as will be discussed subsequently.

Thermal Fatigue Response Test

To check the thermal fatigue response, the following heat treatments were performed on 10 ceramic samples embedded with optical fibers.

Table 1: Heat treatments performed on the samples.

Sample #	Temperature (in C)	Holding Time	# of cycles
1	900	1 hour	1
2	900	24 hours	1
3	900	100 hours	1
4	900	1 min	10
5	900	1 min	100
6	1300	1 hour	1
7	1300	24 hours	1
8	1300	100 hours	1
9	1300	1 min	10
10	1300	1 min	20

Results and Discussion

The main parameters involved in the process are the viscosity of the slurry mixture, the loading rate during pressing, the maximum compressive load, the drying of the green body and the thermal cycle during sintering.
Viscosity plays a significant role in the flow behavior of the slurry while pressing. A low viscosity can lead to the powder being transported with the liquid during pressing, whereas a high viscosity restricts the flow and may lead to formation of voids. Viscosity is determined by the amount of water and binder used. A correct amount of binder can improve the plasticity, reduce the liquid migration rate and improve the green strength. The emulsion used for this process is good for admixing at room temperature, soluble in water and greatly increases the strength of the green body.

The pressing and drying procedures are critical to ensuring uniformity in the density of the ceramic final product. A slow loading rate is essential to allow sufficient time for the slurry to flow and lead to good filling, to avoid pressure gradients and density variation and to eliminate void formation. Sudden decompression or unloading should also be avoided to minimize elastic expansion or springback. The drying of the green body must be carefully controlled to avoid differential shrinkage, which can lead to warping and defects such as cracks in the product. Preliminary drying by natural convection on all faces followed by furnace drying at a very slow rate minimizes differential shrinkage. During the firing process, sufficient time is allowed for binder burnout and a slow rate of heating and cooling is used to avoid excessive thermal stresses. A stress buildup can give rise to warping or

even crack formation. The current schedule for firing results in samples that have no cracks or bending.

The thermal fatigue response tests performed on the finished products comprise an important part of this study. Cracking in the ceramic matrix and debonding between the fiber and matrix are the two major defects that can arise due to thermal cycling. Presence of either of these defects in the sample can result in faulty detection of stress levels in the sample when the optical fiber is used as a stress sensor or can render the sensor completely useless. Any debonding can reduce the level of strain in the fiber and thus lead to an incorrect measure of stress. Table 1 shows the thermal cycling tests that were performed at 900° and 1300° C. At the end of the cycling each sample was carefully examined by both naked eye and an optical microscope with up to 400x magnification. The samples showed no signs of microcracks, debonding or damage to the fiber. This is significant, as it proves the applicability of the sensor in high temperature fatigue environments. Preliminary investigations of these samples by a scanning electron microscope (SEM) is in progress and will be published as soon as it is completed.

SUMMARY AND CONCLUSIONS

Opto-mechanical response of the fiber to external perturbations was examined. The results indicate that the combination of the spatial modulation technique with multimode sapphire fibers appears to be an attractive methodology for the development of optical sensors for very high temperature applications. Based on these preliminary investigations, this technique has been shown to be a sensitive, simple, and cost effective tool for high temperature sensor applications. Optical sapphire fibers can be employed as intrinsic sensors for on-line monitoring of ceramic composites as well as extrinsic sensor for displacement measurements.

In addition, a processing methodology to embed optical fibers in alumina matrix for intrinsic sensor applications has been developed. The process involves the standard steps of slurry preparation, pressurized casting and sintering. The key issues such as the viscosity and concentration of the slurry, loading rate and maximum compressive load during pressing, prevention of bending and damage to the leads of fiber, and sintering cycle were investigated. The correct combination of these parameters have been determined.

The thermal fatigue response studies comprised an essential part of this investigation. The absence of cracks in the matrix or debonding between the optical fiber and matrix proves the feasibility of using this kind of sensor in high temperature environments.

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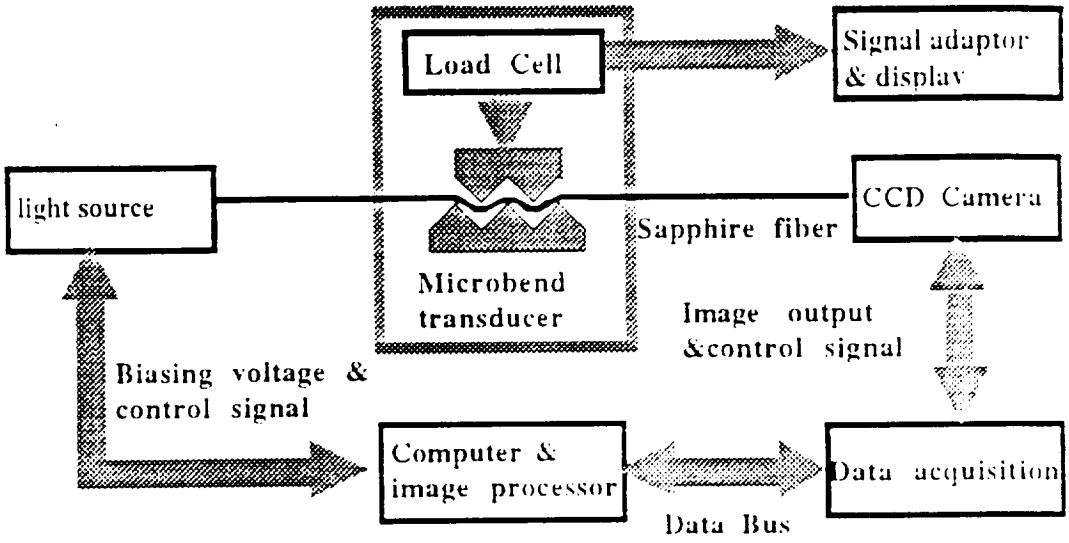

Figure 1. A general block diagram of the developed experimental set-up for measuring the sensor optical response for micro-bending stresses.

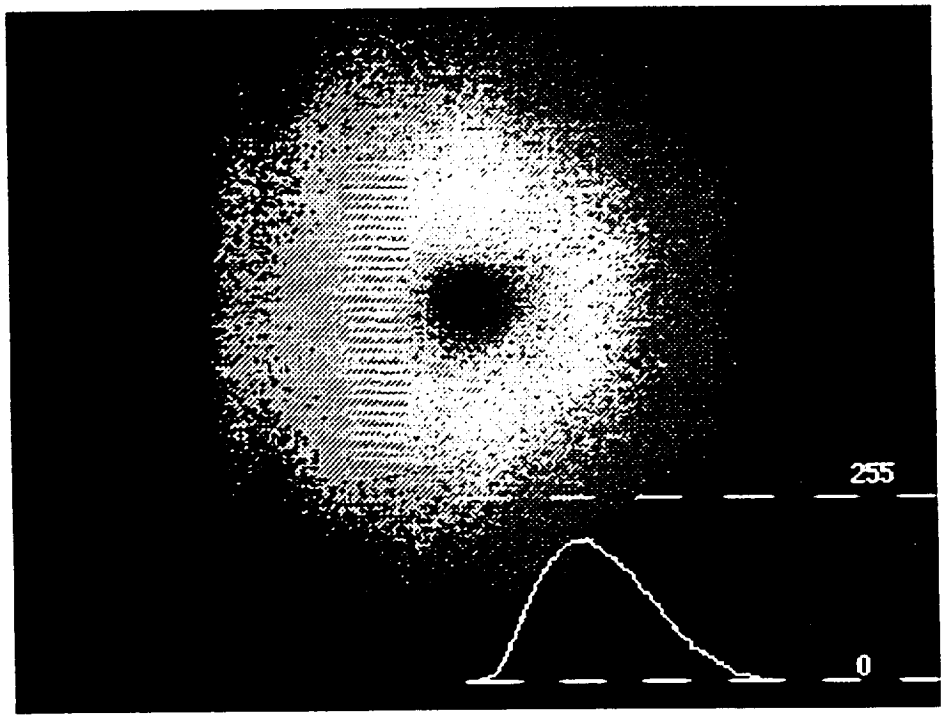

Figure 2. The 2-D image of the far-field pattern and horizontal intensity profile for a sapphire optical fiber before bending was applied.

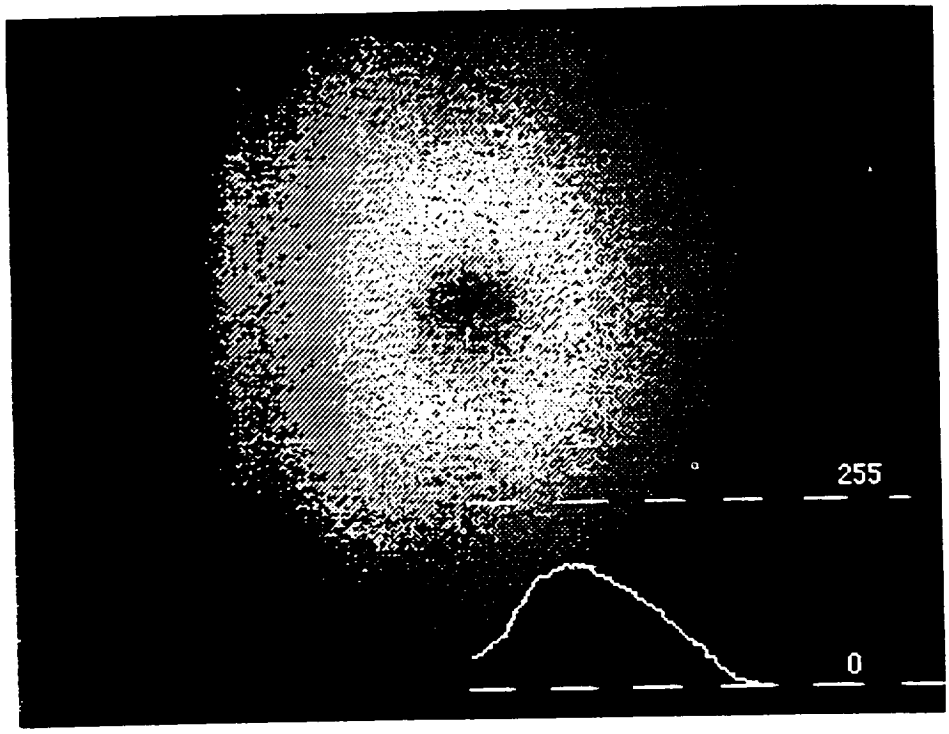

Figure 3. The 2-D image of the far-field pattern and horizontal intensity profile for a sapphire optical fiber under bending test when the vertical dispiacement was about 7 μm.

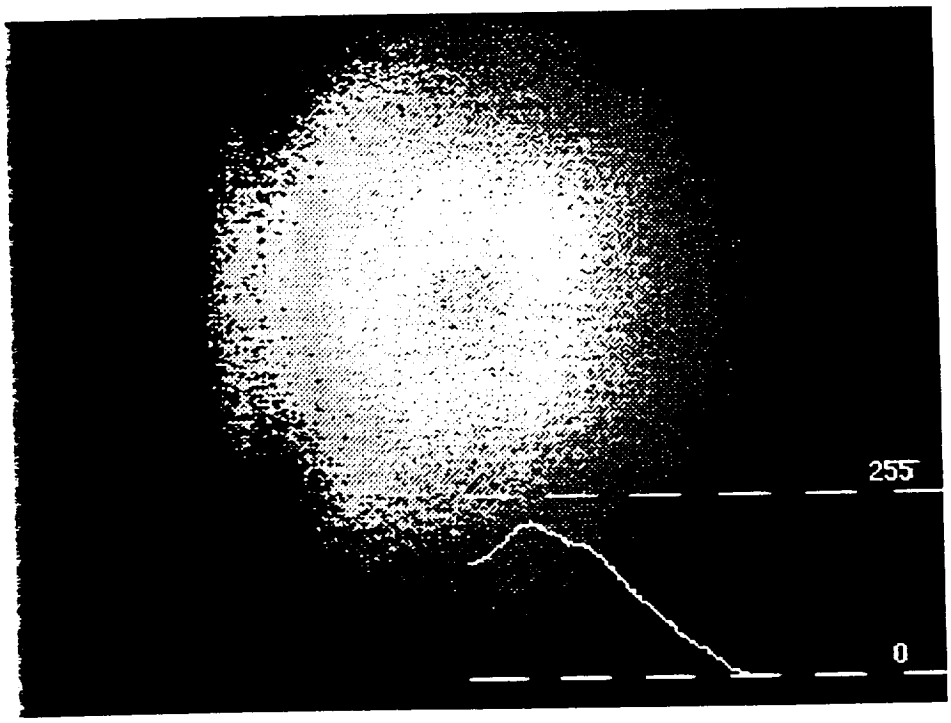

Figure 4. The 2-D image of the far-field pattern and horizontal intensity profile for a sapphire optical fiber under bending test when the vertical dispiacement was about 10.5 μm.

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A NOVEL SAPPHIRE FIBER-OPTIC SENSOR FOR TESTING ADVANCED CERAMICS

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ABSTRACT

The development of an optical sapphire waveguide for high temperature applications is presented. An optical grade sapphire fiber was used as the core of the waveguide. This fiber was coated with a layer of polycrystalline alumina to construct the cladding layer of the waveguide. This optical waveguide can be coated with a protective layer such as silicon carbide and used as an intrinsic sensor for on-line testing of ceramic materials. Preliminary investigations with this approach appear to be very promising in terms of confining optical signals within the fiber core.

INTRODUCTION

The development of high temperature materials for the 21st century will involve novel high temperature test methods in order to simulate a given material's performance under operating conditions. One proposed test method involves using embedded sapphire fiber-optic sensors to measure material damage and stress distribution. Generally, this concept may be useful to: (a) evaluate nondestructively at room-temperature the extent of residual stress state in the composite caused by high-temperature service conditions, and (b) allow continuous in-situ monitoring of the composite performance under end use environments.

Conventional optical fibers can be classified according to their propagation characteristics as single-mode, dual-mode or multimode fibers. The core diameter of a single-mode fiber is in the range of 6-10 μm, and can only support a single mode of propagation within the optical fiber. On the other hand, multimode fibers have a core diameter of 25-200 μm or more and can support hundreds (or even thousand) of propagating modes. Single-mode (or dual mode) optical fibers, made of silica or polymer based materials, have been proposed as embedded sensors for testing composite materials at room temperature. These sensors operate on the

principle of phase and polarization modulation techniques. At temperatures over 200°C, only silica fibers are applicable. At very high temperatures (over 1000°C), sapphire fibers offer an alternative technology for sensor applications. Sapphire (Al_2O_3) fibers possess reasonable optical propagation properties and can potentially perform without significant degradation up to 1500°C (2732 F). However, the difficulties of producing single-mode or dual-mode sapphire fibers limit their application as intrinsic embedded sensors, and most of the reported research employed optical sapphire fibers as extrinsic sensors [1,2]. To overcome these limitations, a novel modulation technique applicable to multimode sapphire fibers is required.

In previous work, an ordinary multimode silica fiber was examined as an embedded sensor for testing polymer matrix composites. For this embedded sensor, a novel spatial modulation (SM) technique was applied and experimental investigation was successfully carried out [3,4]. Analysis and experimental results indicate that the developed SM technique is inexpensive, highly sensitive and well suited for testing composite materials [5,6]. In order to optimize the optical sensor for high temperature ceramics, a modified multimode sapphire fiber is proposed for this type of application.

In this paper, the development of a protectively-coated sapphire multimode optical fiber is presented. This fiber will be used as the sensing element of an embedded sensor. The principle of operation of this sensor is based on spatial modulation and will be discussed in the next section. For experimental investigation, an optical grade sapphire fiber was used as the core of the sensing element; this fiber was coated with cladding and protective layers. The quality of interface contacts and adhesion between successive layers in sapphire optical waveguides are very important, but available deposition techniques may not provide the necessary quality. Therefore, a novel chemical deposition technique, developed at Drexel University in collaboration with the NASA Lewis Research Laboratory, was applied and experimentally tested. The optical fiber was coated with a thin layer of polycrystalline alumina by introducing very fine alumina particles (20 nm) in a polymerizable monomer carrier. The monomer was allowed to polymerize, leaving a thin coating layer on the fiber surface. This thin layer of fine particles provides the necessary bonds between the optical fiber core and the cladding material. Binder removal and subsequent sintering was performed by heating the coated fibers to 600°C and ~1400°C, respectively. In order to reduce the number of interfaces in the structure of the multilayer fiber, the same deposition technique was repeated to grow the cladding layer directly on the fiber core. In this way, the necessary requirements for strong bonds with the fiber core was achieved. Several samples of single crystal sapphire fibers were coated and examined by a scanning electron microscope (SEM).

SENSOR OPERATING PRINCIPLES

The principle of operation of the developed optical sensor is based on spatial modulation (SM) of optical signals propagating through a multimode fiber. Within a multimode optical fiber, optical signals propagate according to the modal structure of the fiber and the boundary conditions. Altering the boundary conditions of an

optical fiber induces modal coupling and results in modal power distribution (MPD) modulation. The Coupled-Mode-Theory can be employed for the analysis of the MPD modulation [7]. The application of the spatial modulation technique in optical sensors has been successfully tested in embedded sensors, for real-time characterization of smart structures/materials [8]. Deforming the embedded fiber by mechanical stresses or other forms of perturbation results in MPD modulation which can be exploited to sense the applied signals [5,6]. The measurements of the distribution and subsequent redistribution of the modal power can be accomplished by scanning the far-field pattern at the fiber end using a CCD camera or array of photodetectors. The experimental set up developed for opto-mechanical measurements is shown in Fig.1.

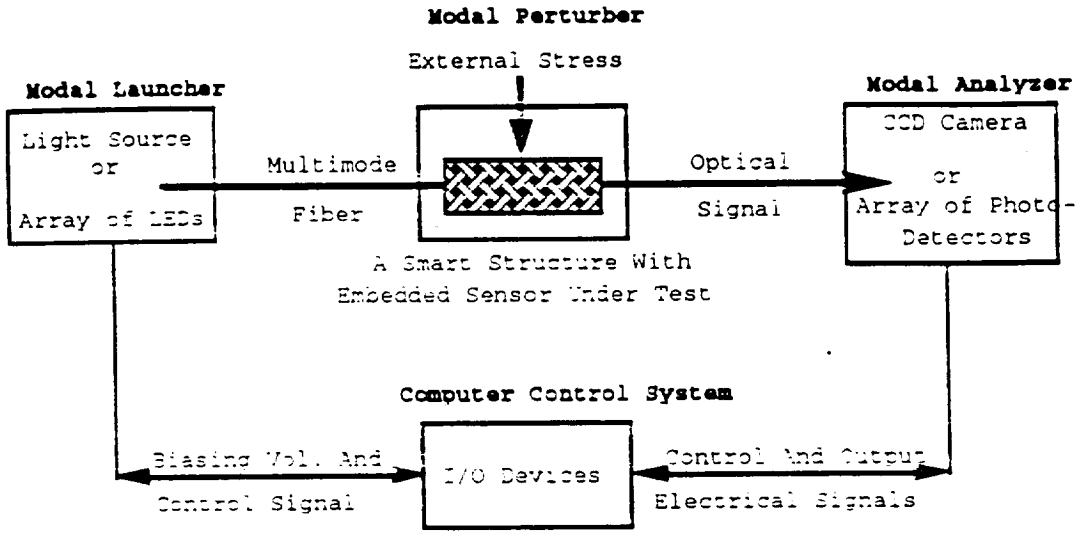


Fig.1. A general block diagram of the developed characterization method, where (a) the modal launcher is an optical source or array of sources used to excite a limited group of modes within the optical fiber, (b) the modal perturber is the embedded fiber-optic sensor under test, (c) the modal analyzer is the detection system of the modal power positioned at the output end of the optical fiber. All are under computer control.

The sensitivity of this type of embedded fiber-optic sensor is related to the modal structure of the fiber and to the mechanical behavior of both the fiber and the materials surrounding it. The level of sensitivity is strongly dependent on the elastic modulus of the surrounding materials and the fiber coating. It was shown that the sensitivity of a developed embedded sensor employing the MPD technique was enhanced by accurately exciting a limited number of higher order modes and using a proper optical fiber coating [9].

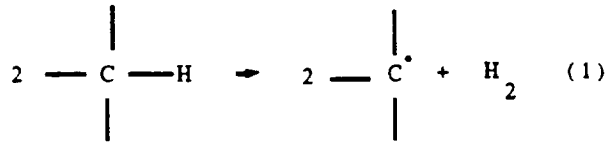
OPTICAL WAVEGUIDE DESIGN AND FABRICATION

Design and Coating Requirements

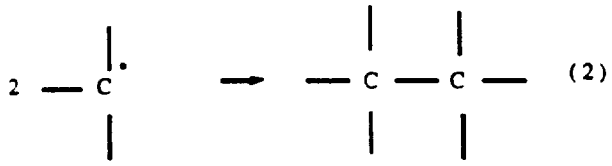
Optical grade sapphire fibers can be grown as high quality alumina single crystal fibers. In contrast to conventional silica based optical fibers, which have a cladding

layer of a lower refractive index than the core materials, these alumina fibers are unclad. Cladding layers are required to confine the propagating optical signal within the fiber core and limit attenuation and losses. In addition, an external protective coating is needed to protect the embedded fiber from its surrounding environment. Therefore, two layers of coating on the sapphire fibers are necessary. These two layers will confine the optical signals and protect the fiber over a wide range of temperature, pressures and environmental conditions. For thermal durability of the sapphire optical waveguide, alumina is the preferred cladding material. The thickness of the cladding layer should be on the order of 20-30 μm. For the second layer of coating, the protective layer, silicon carbide (SiC) is preferred to other materials due to its mechanical and thermal properties. A thin layer of SiC grown on top of the cladding layer will prevent long term deterioration of strength and stiffness of the optical fibers at elevated temperatures. This layer will also limit the sources of surface damage existing at the matrix/fiber interface.

A chemical deposition technique was developed for the surface treatment of the optical grade sapphire fiber. The fiber was coated with a thin layer of polycrystalline alumina by introducing very fine alumina particles (20 nm) in a polymerizable monomer carrier. The polymer is compatible with the sapphire fiber surface, giving good adhesion to both the particles and the fiber surface. In polymerization, chemical activators are used to promote polymerization at room temperature. Currently, most chemical polymerizations utilize an amino-peroxide initiating system (for example using the accelerator N-Ndimethyl-para-toullidine [DMPT] and the initiator benzoyl peroxide [BPO]). DMPT is added to the monomer (acrylic acid) and interacts with BPO. The product is added to the filler (fine particles of alumina powder), to produce free radicals on the polymer side chains. These radicals, by saturating one another, produce C-C crosslinks. Thus, the cross linking in its simplest terms can be expressed as follows:



(1)



(2)

However, the cross linking mechanisms can sometimes be more complex. In various suspension systems, 0.05% to 2.0% of the initiator DMPT and 0.5% to 2.0% the accelerator BPO are used for the monomer polymerization, based on the weight of the monomer. The coating thickness can be controlled by varying the quantities of the initiator and the accelerator to adjust the working time of the suspension before hardening. This technique was used for surface treatment and repeated for coating the cladding layer.

Preparation of Homogeneous Suspension

According to the coating requirements discussed before (specifically, coating the fiber with 20-30 μm alumina cladding), a suspension of about 0.05 gm of both DMPT and BPO were added to 10 ml of acrylic acid and 10 ml of H_2O. The BPO was dissolved in acetone before being added to the mixture. The mixture was allowed to dry at room temperature to remove the acetone. Very fine aluminum oxide (Al_2O_3) particles of 20 nm diameter were added to the mixture at different loading levels (1, 1.5, 2, and 2.5 gm). A spinner was used in order to promote homogeneity in the solutions.

Surface Treatment and Coating of the Fiber Core

The 0.125 mm diameter optical grade sapphire single crystal fibers used in these experiments were grown at the Massachusetts Institute of Technology (MIT) using a laser heated fiber growth process [10]. There are several critical parameters involved in the coating process. Therefore, a large number of these optical grade sapphire fibers were prepared and coated at different conditions. These optical sapphire fibers were cleaned by hydrochloric acid (HCl concentration 36%) and washed in distilled water. Then each fiber was coated with a thin layer of fine particles to provide the necessary bonds between the optical fiber core and the cladding material. Each fiber was immersed in a prepared suspension containing 10%, 15%, 20%, or 25% aluminum oxide, for half an hour, resulting in the deposition of a thin layer of the coating on the fiber surface. These fibers were left for 24 hours to polymerize in room temperature. Then the coated fibers were heated to 600°C for binder removal. The time needed for complete polymer evaporation depends on the thickness of the coating layer and the materials concentrations. Therefore, different times (10-60 minutes) were used in the interest of completeness. The temperature was then increased for each fiber to a different degree between 1300°C and 1500°C for varying time duration's (10-60 minutes), in order to sinter the alumina powder around the fiber. The heating and cooling rates for the furnace were in the 2-10°C/min range. The same procedure was repeated with different mixture concentrations for coating the cladding and the protective layers. The thickness of the cladding layer was controlled by changing the deposition rate (changing the concentration of the materials in the mixture and/or changing the deposition time). Each of the samples was examined and the results are presented next.

RESULTS AND DISCUSSION

At each stage of the process, the surface morphology of the coated fibers were examined and studied using a scanning electron microscope (SEM). At first, the prepared optical grade sapphire fibers were examined by SEM before coating. The results show a very smooth surface with negligible variation in the fiber diameter. Then the samples were studied after coating. It was clear that the coated layers were not homogeneous and/or the sintering process was not completed in the majority of these samples. For these samples, the alumina concentration in the

solution might have been low, or the heating temperature or time may not have been sufficient. Another major observation made was that diffusion started at 1500°C. As shown in Fig. 2, the surface morphology of the fiber started to change at 1500°C. Accordingly, the maximum temperature used for our experimental investigation was limited to 1400°C.

One the other hand, the results show that a few of these samples were perfectly coated. It is encouraging, however, to find that the samples with 20% concentration of alumina produced the highest level of uniformity and homogeneity. Several additional experiments with this coating concentration were prepared and the results were consistent. For these samples, the fibers were heated to 600°C for one hour to ensure that the polymer was completely removed, and the temperature was then raised to 1400° C for an hour for perfect sintering of the alumina powder on the surface of these fibers. As shown in Fig. 3, a thin layer of homogeneous alumina is coated on the surface of the optical fiber. Finally, in order to coat the cladding layer to a thickness of about 20 μm, the fibers were coated with three layers of the prepared suspension in sequence before they were heated. The thickness of the cladding layer was controlled by changing the concentration of the materials in the suspension. The result is shown in Fig. 4. In this manner, a high quality cladding layer was successfully obtained. Preliminary optical tests show good confinement of the optical signal within the fiber core.

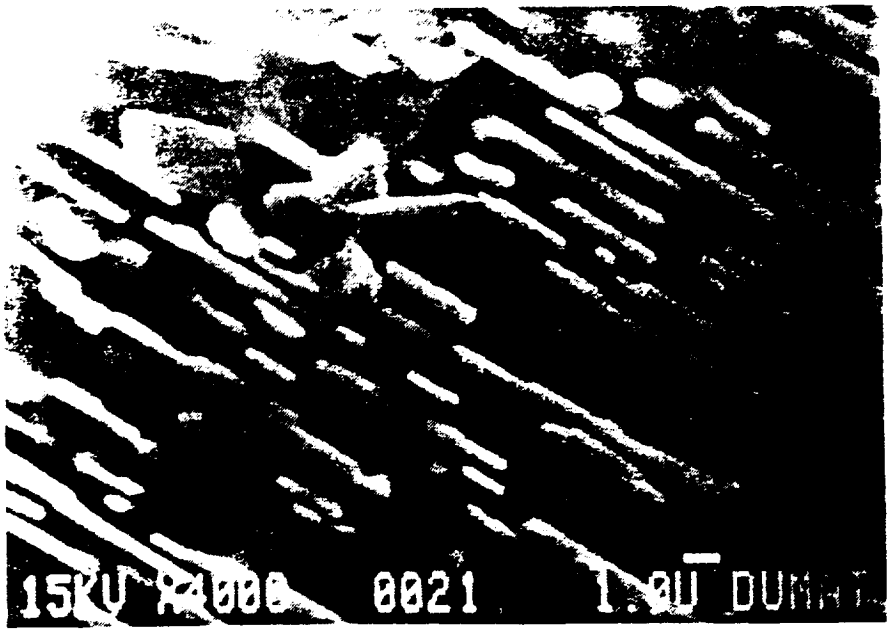


Fig. 2. The SEM result, for a sapphire fiber treated with suspension having 20% alumina concentration, after the binder was removed the fiber was heated at 1500°C for sintering the alumina.(Magnified to 4000x).

Fig. 3. The fiber was coated with a thin layer of alumina, using a suspension having a 20% alumina concentration and followed by thermal treatment at 600°C for one hour for binder removal and at 1400°C for an hour for sintering the alumina (Magnified to 4000x).

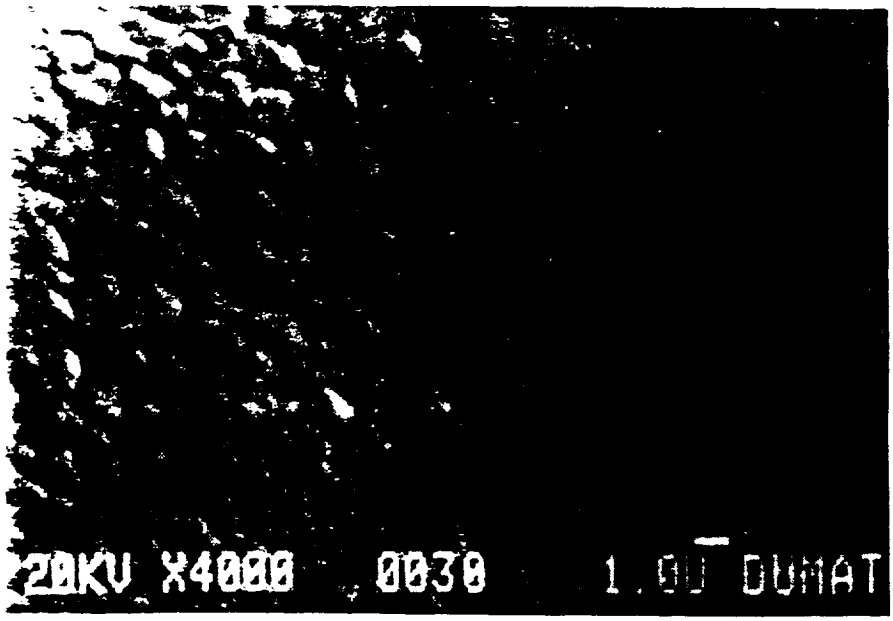


Fig. 4. The fiber was coated with three layers to construct the cladding layer (Magnified to 4000x).

CONCLUSION

A chemical deposition technique was developed and tested for the homogeneous coating of sapphire fibers. SEM study showed that a uniform layer of polycrystalline alumina was coated on the surface of the fiber core. During the course of the study, several difficulties arose in determining the key parameters required to optimize the process. The highest level of uniformity and homogeneity was achieved by using a suspension containing 20% alumina, and a maximum sintering temperature of 1400°C.

The coating technique developed in the study is crucial for the fabrication of reliable optical waveguides for a broad range of fiber-optic sensors. Initial tests of the opto-mechanical behavior of these sensors were encouraging and they will form the basis for future publications.

ACKNOWLEDGMENT

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Optical Response of Sapphire Multimode Optical Sensor for Ceramic Composite Applications

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ABSTRACT

The main objective of this study is the development of an embedded fiber optic sensor for testing ceramic composites in a very high temperature environment. The sensing element is an optical grade sapphire fiber operating on the principle of spatial modulation in a multimode waveguide. In order to employ this waveguide as a stress sensor, opto-mechanical testing has been performed to examine the optical response to external stresses. Several tests, including tension, micro-bending, and lateral compression, are in progress. These tests will establish the basis for using embedded optical sensors for characterization of ceramic composites in real environment. The principles of operation and experimental investigations on the microbending tests are presented in this paper. The results show that the developed sensor can be applied for stress monitoring as well as displacement measurements in a very high temperature environment.

1. INTRODUCTION

Embedded fiber-optic sensors offer significant potential for application in composite materials. Optical fibers made of silica or polymer based materials have been proposed as a sensing element for testing composite materials at low temperatures.[1] Only silica fibers are usable for temperatures over 200°C. At very high temperatures (over 1000°C), optical grade sapphire fibers offer an alternative means for sensor applications. Sapphire (Al_2O_3) fibers possess reasonable optical propagation properties. However, the difficulties of producing single-mode fibers limit their application to phase (interferrometric) or polarization modulation techniques. This limitation has directed our research to a spatial modulation (SM) technique, previously developed and tested with conventional amorphous multimode fibers.[2,3]

Available optical grade sapphire fibers are unclad single crystal fibers. Two layers of coating on the surface of these fibers are necessary; a cladding layer is required to confine the optical signal within the fiber core, and a protective coating is needed to protect the embedded fiber from its surrounding environment. A chemical deposition technique was developed for coating these fibers. The fibers were coated with a thin layer of polycrystalline alumina by introducing very fine particles (20 nm) of the material in a

polymerizable monomer carrier used as a suspension. The suspension was prepared of 0.05 gm tertiary amine accelerator N-Ndimethyl-para-toullidine (DMPT) and 0.05 gm benzoyl preoxide (BPO) initiator added to 10 ml acrylic acid monomer and 10 ml H_2O. Aluminum oxide (Al_2O_3) particles were added to the suspension and the powder was mixed well to have a homogeneous solution. The fibers were immersed in the prepared solution for half an hour. Then, the fibers were left for 24 hours to polymerize in room temperature. The coated fibers were then heated to 600°C for binder removal. The time needed for complete polymer evaporation depends on the thickness of the coating layer and the materials concentrations. The temperature was then increased to 1400°C in order to sinter the alumina powder on the fiber surface. For coating a 20-30 μm cladding layer, the same procedure was repeated several times. In this way, the necessary requirements for strong bonds with the fiber core was achieved. A detailed description of this process is reported elsewhere.[4]

A protective thin layer of silicon carbide was then applied using a plasma enhanced chemical vapor deposition (PECVD) technique. The pressure of the reactor was maintained below 200 mtorr, and the parallel plate electrodes were connected to a 13.56 MHz rf supply. The substrate holder was maintained at a constant temperature by a recirculating cooler. The feed gas consisted of a mixture of tetramethyl silane and hydrogen. Adjustable deposition parameters include chamber pressure, gas ratio, power level, and substrate temperature are varied to obtain films with desired properties. These parameters were adjusted for deposition of a uniform 0.25 μm silicon carbide layer on the top of the cladding layer. In this manner, a high quality coating was successfully obtained.

In order to use these optical grade sapphire fibers for sensor applications, the fiber response to external perturbations or stresses must be evaluated. In this paper, the fiber response to microbending test is presented. A 5-point bending transducer was used to induce sinusoidal displacement on a small part of the fiber. A load cell was used to vary the amplitude of the sinusoidal displacement. At the optical fiber receiving end, a CCD camera was used to detect the 2-D optical intensity modulation induced by displacement variations. The principle of operation and the experimental results are presented next.

2. PRINCIPLE OF OPERATION AND EXPERIMENTAL SET-UP

Within a multimode optical fiber, optical signals propagate according to the modal structure of the fiber and the boundary conditions. Altering the boundary conditions of an optical fiber induces modal coupling and results in modal power redistribution within the fiber core.[2] Therefore, the sensor's operating principles are based on spatial modulation (SM) of optical signals propagating through multimode fibers. The modulation is two dimensional across the fiber cross section. The Coupled-Mode-Theory can be employed for the analysis of the modal power distribution MPD modulation.[5]

In previous work, the spatial modulation technique has been used to detect external stresses applied to silica fibers embedded in polymer composites.[6,7] External stresses applied to the structure result in modal power redistribution within the optical fibers. The measurements of the distribution and subsequent redistribution of the modal power were exploited to sense applied stresses. These measurements were accomplished by scanning the far-field pattern at the fiber end. A signal processing and analysis program having a special imaging algorithm was used. Preliminary tests show that this imaging algorithm

is compatible with the SM technique and capable of providing a detailed structure of the fiber far-field pattern in three dimensions. Analysis and experimental results indicate that the developed SM technique is inexpensive, highly sensitive and well suited for multimode fiber-optic sensors.[8]

In order to optimize the developed sensor for high temperature applications, it was proposed to use optical grade sapphire fibers to replace silica fibers within the sensor. The experimental set-up developed for previous work was modified for measuring optical response to microbending (Fig. 1). A light source was used to excite a limited group of modes within the optical fiber. A modal perturber, consisting of a load cell and a 5-point bending transducer, was used to apply quantified sinusoidal displacement to a small portion of the optical fiber under test. The 5-point bending transducer consists of two metal plates. The lower plate is stationary and has three equally spaced ridges, 0.9 cm apart, while the upper plate is mobile and has two ridges 0.9 cm apart. The load cell, in connection to a vertical shaft, is used to apply vertical displacement to the upper plate. At the output end of the fiber, the far-field pattern emerging from the fiber was scanned by a high-sensitivity and high-resolution CCD camera. The camera output was monitored through a data acquisition and an image processing board installed in the computer controller. This board contains a frame grabber and digitizer, enabling video freeze frame and access to the gray level of each pixel. Special algorithms were used to filter out interference noise and improve the signal to noise ratio. This experimental set-up, shown in Fig. 1, was used to examine the response of sapphire fibers to 5-point bending tests.

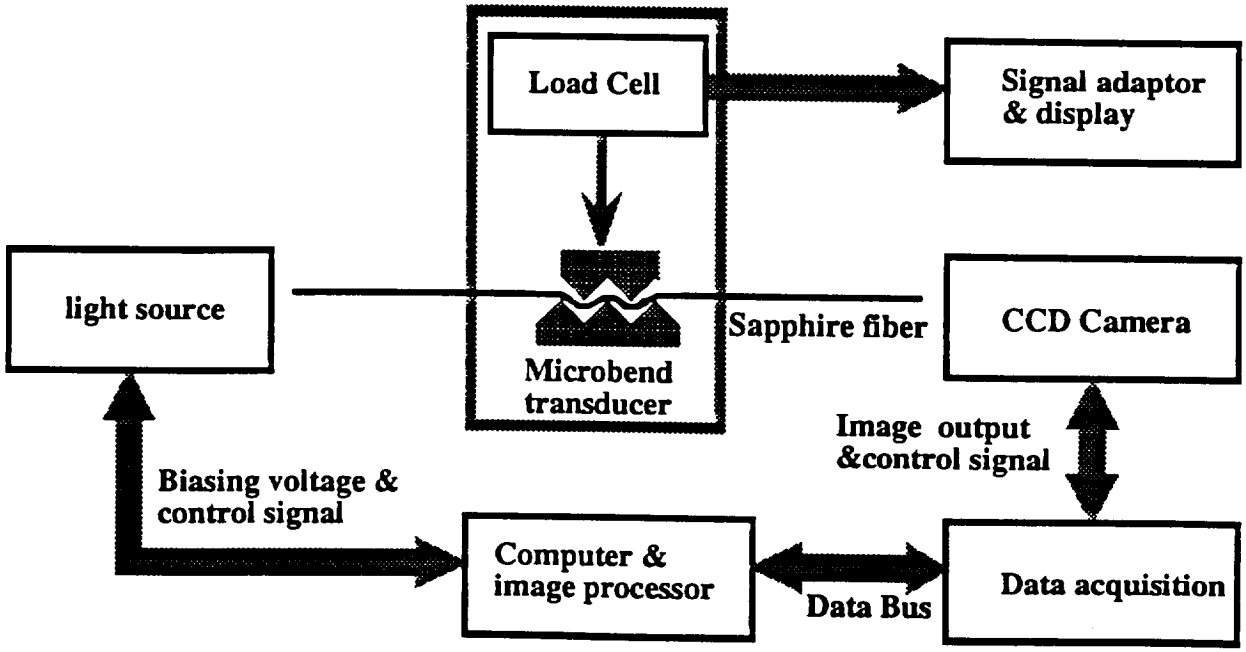

Fig. 1. A general block diagram of the developed experimental set-up, for measuring the sensor optical response to microbending stress.

3. EXPERIMENTAL INVESTIGATION AND RESULTS

Several sapphire fibers were prepared and tested under various bending levels. The fibers were first cleaved and the end faces perfectly polished. The light launching angle used during this experimental investigation was about 8 degrees. These launching conditions were used to excite higher order modes which are more sensitive to external perturbations than lower order modes. The external perturbations were applied in the form of sinusoidal 5-point bending. The amplitude of the sinusoidal displacement was measured and the far-field pattern was scanned for different bending levels.

The bending effect on modal power distribution (MPD) was predicted for different amplitudes of the sine-wave displacement by scanning the far-field pattern. Several measurements were taken while increasing the amplitudes of the sine-wave bending, and during decreasing this displacement. Preliminary results show that there is not much difference in the results obtained while increasing or decreasing the same bending geometry. 3-D image processing algorithms were used to enhance, analyze, edit, and display the far-field pattern. As well as predicting the far-field pattern profile (MPDs) for each scan.

Samples of the results obtained for an optical grade c-axis sapphire fiber prepared by the Edge Fed Growth Puller technique are shown in Figs. 2-5. The first figure (Fig. 2) shows the 3-D far-field pattern recorded at the fiber output, before any stress was applied (i.e. the MPD with no applied bending [initial state]). The fiber was excited with an optical beam at an angle 8^0 off-axis. The normalized output intensity profile of the far-field pattern vs. the scanning angle is also shown on the same figure. It is clear from this figure that the crystallographic structure of the fiber has resulted in a radial periodic interference pattern. However, the effect of interference noise was reduced by using special image processing algorithms. Also, this figure shows that optical intensity for the lowest order modes (on-axis modes) is almost zero, and most of the power is concentrated around an angle equal to the excitation angle.

Due to the crystallographic structure of the fiber (as opposed to the amorphous structure of the silica fibers) the pattern of the excited modes shown in Fig. 2 was broader than that obtained before with amorphous silica fibers, under similar conditions.[8] This is due to diffraction and interference of optical signals by the crystal planes. Regardless of the crystal effect the MPD technique was applied to the fiber and successfully detected changes in the far-field pattern induced by external stresses. Special signal processing algorithms were used to limit the crystallographic structural effect and to enhanced the far-field pattern. In addition, these algorithms were used to dramatically improve the signal to noise ratio, which will enhance the sensitivity of the sensor. Samples of the micro-bending results are presented next.

In the presence of a small displacement (undulation) applied to the optical fiber, an induced change in the MPD was detected, as shown in Fig. 3. The amplitude of the sinusoidal displacement recorder for this far-field pattern was 3.5 μm. The applied perturbation causes an intermodal coupling, leading to a modal power redistribution, which is broadened toward smaller angles reflecting a transfer of power to lower-order modes.

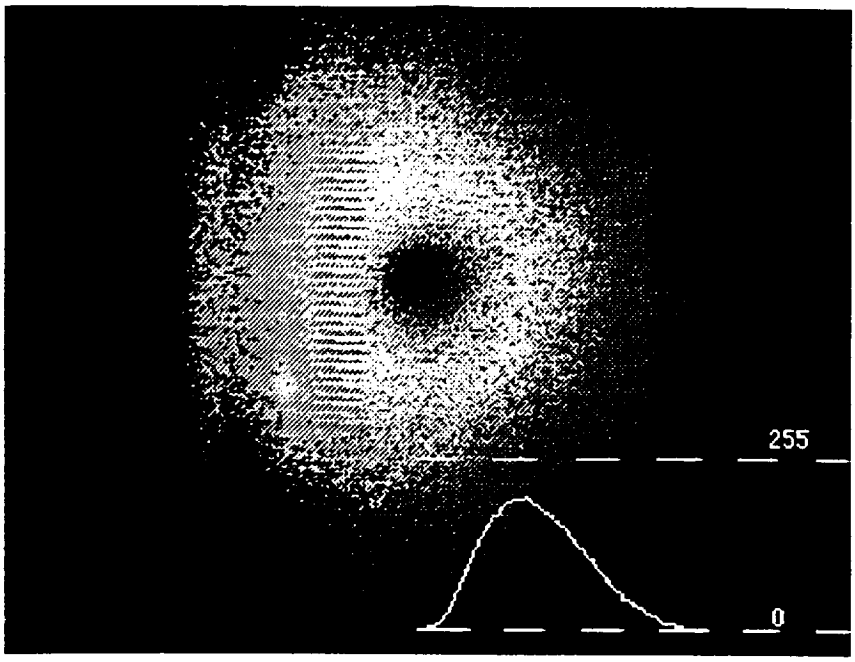

Figure 2. The 2-D image of the far-field pattern and the horizontal intensity profile for a sapphire optical fiber before bending is applied.

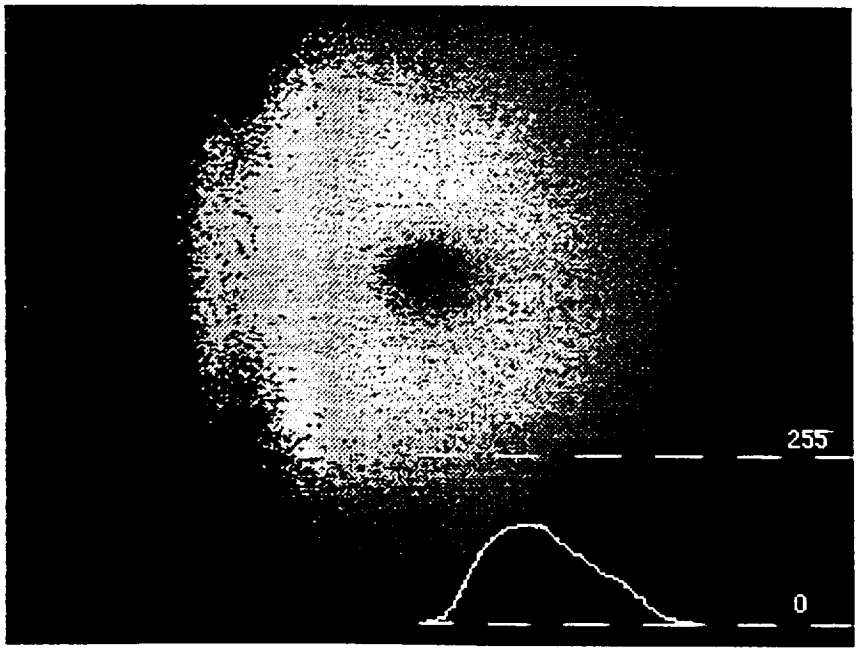

Figure 3. The 2-D image of the far-field pattern and the horizontal intensity profilefor a sapphire optical fiber under bending test when the vertical displacement is approximately 3.5 μm.

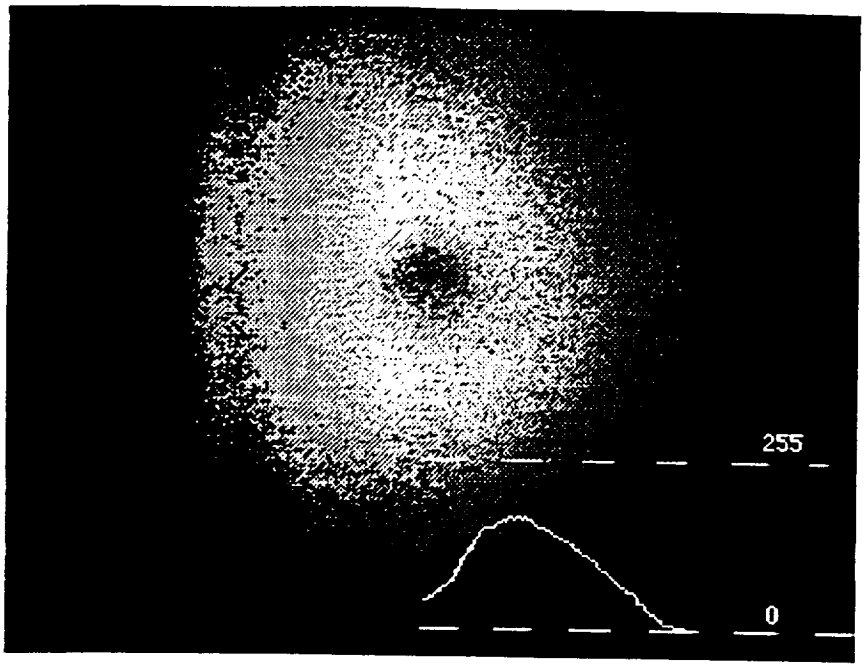

Figure 4. The 2-D image of the far-field pattern and the horizontal intensity profile for a sapphire optical fiber under bending test when the vertical displacement is approximately 7 μm.

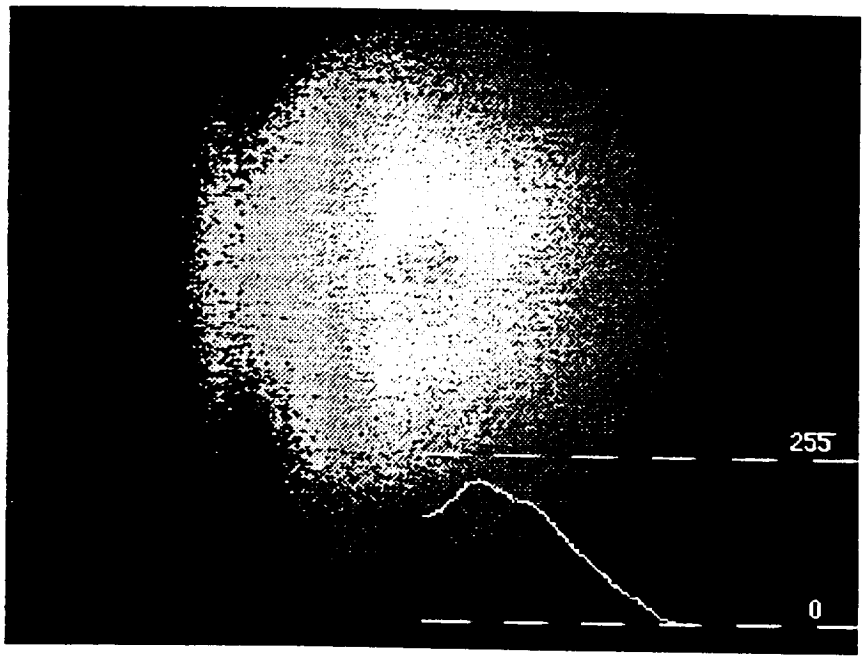

Figure 5. The 2-D image of the far-field pattern and the horizontal intensity profile for a sapphire optical fiber under bending test when the vertical displacement is approximately 10.5 μm.

The sensitivity of the sensor, shown in figure 3, is high enough for displacement measurements. It can be improved by varying the geometry of the 5-point transducer. As applied undulation was increased (from 3.5 μm to 7 μm), considerable rearrangement of the modal power was shown (Fig. 4). This bending level causes more intermodal coupling, leading to a considerable modal power redistribution, which is broadened toward smaller angles reflecting a transfer of large optical power to lower-order modes. For higher bending (9 μm), more power transferred to the lower order modes. As the amplitude of the sinusoidal undulation increases, much more power is shifted to the center of the pattern for a bending amplitude about 10.5 μm (Fig. 5). However, the change in the total optical output power at each of these bending levels was still negligible. Further increase of the applied bending (over 20 μm) results in small optical loss and decrease of the total throughput. This indicates that measurements on modal power distribution are much more sensitive than total intensity modulation for this type of application.

This experimental work was focused on the application of the modal power variation within optical sapphire fiber under various bending conditions. Specifically, it was directed toward experimental evaluation of MPD for lower order modes in response of undulation (i.e. changes in the optical intensity around the center of the far-field patter). Accordingly, based on the results shown in Fig. 2-5, a simple relation was drawn for the on-axis intensity of the far-field pattern in response to the amplitude of sinusoidal undulation (Fig. 6). This figure shows a uniform smooth variation in optical intensity at the center of the fiber induced by changes in the bending level. As a result of this simple correlation a direct approach and cost effective methodology is suggested. In this approach, the CCD camera can be replaced by a single photodetector. This detector will be positioned on the fiber axis to collect only the power of the lower order modes. The output of this detector can be coupled to a simple biasing circuit to indicate the change % in the optical intensity output, at the center of the far-field pattern. Additional photodetector, positioned at different angle from the fiber axis, can be used to provide a reference signal. The correlation function between the signal intensity profile and the applied stress has not been fully developed yet. This function will be derived in future work.

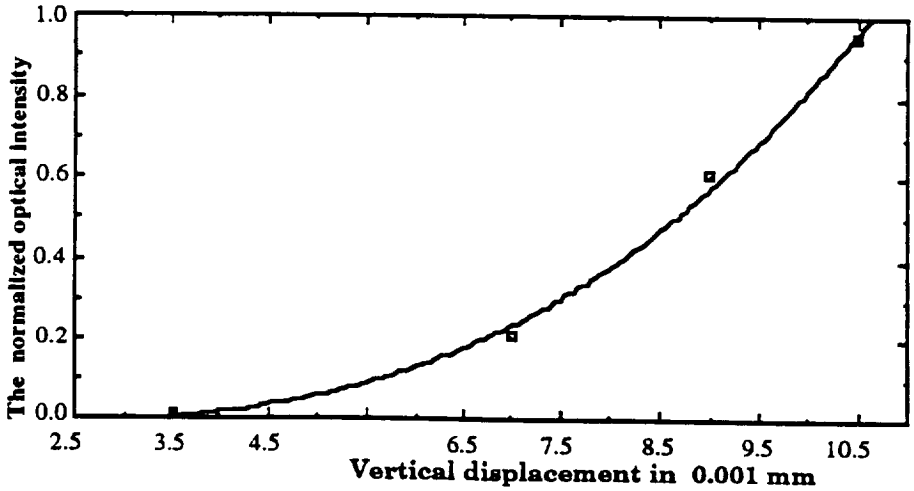

Figure 6. Normalized optical intensity measured at the center of the far-field pattern vs vertical displacement in bending test.

4. CONCLUSION

The combination of spatial modulation with multimode sapphire fibers appears to be an attractive methodology for the development of optical sensors for very high temperature applications. Based on these preliminary results, this technique has been shown to be a sensitive, simple, and cost effective tool for sensor application, and optical sapphire fibers can be employed as intrinsic sensors for on-line monitoring of ceramic composites, and as extrinsic sensor for displacement measurements.

5. ACKNOWLEDGMENTS

This work was supported by the Structural Integrity Branch, NASA Lewis Research Center, Cleveland, OH. It is also supported by the Commonwealth of Pennsylvania Ben Franklin Technology Center. The optical grade sapphire fiber used for this study was provided by the Advanced Crystal Products Corporation, MA.

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The Technical Annual Report

of

A Research Grant Award

1. **Number:** NAG3-1347 **Basic Award**

2. **To:** Drexel University
Philadelphia, PA 19104

3. **For research entitled:**

"Thermal Durability of Sapphire Optical Waveguides Processed Into High Temperature Ceramic Composites."

4. **Under the direction of (Principal Investigator):** Dr. M.A. El-Sherif

5. **Duration:** One (1) Year, Followed by 6 months no cost extension

6. **Beginning date:** April 1, 1992

7. **NASA Technical Officer:** Dr. Don Roth

Address:

Structural Integrity Branch
NASA Lewis Research Center
Mail Stop 6-1
21000 Brookpark Road
Cleveland, OH 44135

8. **Date of report:** July 10, 1993

CONTENTS

Thermal Durability of Sapphire Optical Waveguides Processed Into High Temperature Ceramic Composites

Executive Summary

The development of high temperature materials for the 21st century will involve novel high temperature testing methods in order to simulate a given material's performance under operating conditions. In this project, a novel sapphire fiber-optic sensor has been proposed for this type of application.

The first year work plan was successfully performed. Protectively-coated sapphire multimode optical fibers were designed, fabricated and tested. The fibers were coated with two layers: (a) a cladding layer of polycrystalline alumina, and (b) a protective layer of silicon carbide. A novel chemical deposition technique has been developed to provide a homogeneous high quality coating of the cladding layer. A thin film layer of silicon carbide was then added using the plasma enhanced chemical vapor deposition (PECVD) technique. A Patent Disclosure is in process through Drexel University regarding the developed process (see Appendix A).

In addition, a novel technique was developed for processing ceramic matrix composites containing the developed optical sapphire fibers. Samples of the optical fibers were embedded in aluminum oxide matrix. This processing technique can eliminate problems of debonding and mismatching. Several specimens of size 2.0 in x 1.0 in x 0.25 in were successfully fabricated, each containing one or two optical fibers embedded parallel to the specimen axis, using the pressure filtration method. These samples were preliminary examined by thermal cycling tests. Three samples were heated to 1400°C for three hour and the process was repeated four times (four cycles). The heating and cooling rates for the furnace were in the 2-10°C/min range. All of the samples were survived without damage or crack.

Finally, a preliminary study has been performed to prove the concept of using the MPD technique with multimode sapphire fibers. A specially designed experimental setup coupled to a data acquisition system and signal processing algorithms was used. Based on results obtained, the combination of the MPD technique with the developed sapphire waveguides appears to be an attractive methodology for the development of optical sensor for very high temperature applications.

Thermal Durability of Sapphire Optical Waveguides Processed Into High Temperature Ceramic Composites

I. Introduction

Fiber-optic sensor offers an advanced technology for continuous monitoring of materials behavior in real-time. The sensitivity and dynamic range of these sensors depend mainly on the propagation characteristics of optical fibers. Conventional optical fibers can be classified according to their propagation characteristics as single-mode, dual-mode or multimode fibers. The core diameter of a single-mode fiber is in the range of 6-10 μm, and can only support a single mode of propagation within the optical fiber. On the other hand, multimode fibers have a core diameter of 25-200 μm or more and can support hundreds (or even thousands) of propagating modes. Single or dual mode optical fibers made of silica or polymer based materials have been used for composite materials characterization at room temperature. These sensors operate on the principles of phase (interferrometric) or polarization modulation. At very high temperatures (over 1000°C), sapphire fibers offer an alternative technology for materials sensing applications.

Sapphire (Al_2O_3) fibers possess reasonable optical propagation properties and can potentially perform without significant degradation to more than 1500°C (2732 F). However, the difficulties of producing single-mode or dual-mode sapphire fibers limit their application as intrinsic (embedded) sensors [1,2], and a novel modulation technique required for the available multimode sapphire fibers. This problem guided our attention to the spatial modulation (SM) technique recently developed by Dr. M. El-Sherif, the project PI, and tested with multimode conventional silica fibers for embedded sensor applications.

The operating principle of these sensors is based on spatial modulation (SM) of optical signals propagating through a multimode fiber. Within a multimode optical fiber, optical signals propagate according to the modal structure of the fiber and the boundary conditions. Altering the boundary conditions of an optical fiber induces modal coupling and results in SM of the modal power within the fiber core. The Coupled-Mode-Theory can be employed for the analysis of the modal power distribution MPD modulation [3,4].

The application of spatial modulation to optical sensors has been successfully tested in embedded sensors, for real-time characterization of composite materials at room temperature. Deforming the embedded fiber by mechanical stresses or other forms of perturbation results in MPD modulation, which can be exploited to sense applied signals [5,6]. The measurements of the distribution and subsequent redistribution of the modal power can be accomplished by scanning the far-field pattern at the fiber end using a CCD camera or array of photodetectors. The experimental set up developed for opto-mechanical measurements is shown in Fig.1.

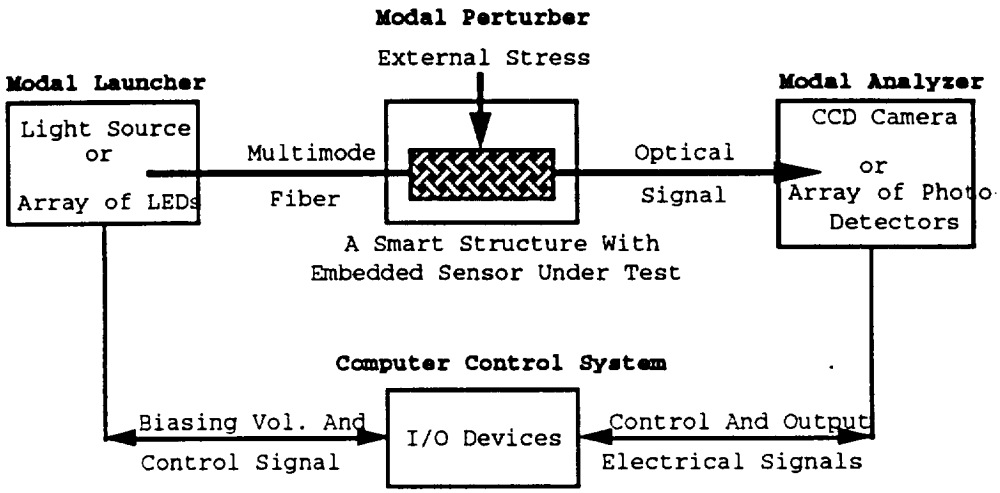


Fig. 1. A general block diagram of the developed characterization method, where (a) the modal launcher is an optical source or array of sources used to excite a limited group of modes within the optical fiber, (b) the modal perturber is the embedded fiber-optic sensor under test, (c) the modal analyzer is the detection system of the modal power positioned at the output end of the optical fiber. All are under computer control.

Sensor sensitivity is related to the modal structure of the fiber and the mechanical behavior of both the optical fiber and the materials surrounding it. The level of sensitivity is strongly dependent on the elastic modulus of the surrounding materials and the fiber coating. It has been shown that the sensitivity of a developed embedded sensor employing the MPD technique can be enhanced by accurately exciting a limited number of higher order modes and using a proper optical fiber coating [8]. The fibers were applied as embedded sensors for testing composite materials at room temperature.

Analysis and experimental results indicate that the developed SM technique is inexpensive, highly sensitive and well suited for multimode fiber-optic sensors [5,6]. In order to optimize multimode optical sensors for high temperature ceramic composites, optical grade sapphire fibers are proposed for this type of application. The combination of spatial modulation with multimode sapphire fibers appears to be an attractive methodology for the development of optical sensors for very high temperature applications.

II. The Work Carried Out

Conventional optical fibers used for transmission of information and sensor applications are made of silica (typically 125 to 140 μm diameter) and are coated with acrylic polymers. For sensor applications, at very high temperatures (over 1000° C), sapphire fibers offer an alternative technology. Sapphire (Al_2O_3) fibers possess reasonable optical propagation properties up to 1500° C (2732 F). In this project, a novel technique is proposed for high temperature sensors, using optical grade sapphire fibers.

During the course of the first year of the project, protectively-coated sapphire multimode optical fibers were designed, fabricated, and tested using the MPD technique. This fiber will be used as the sensing element in optical system. Optical grade single crystal sapphire fibers were coated with two layers; (a) a cladding layer of polycrystalline alumina, and (b) a protective layer of silicon carbide. A novel chemical deposition technique has been used to provide a homogeneous high quality cladding layer [9]. A thin film of silicon carbide coating was applied using the plasma enhanced chemical vapor deposition (PECVD) technique. Preliminary results with this approach appear to be very promising in terms of confining the optical signals within the fiber core and providing a sensitive waveguide for the application of the MPD technique [10,11].

In addition, samples of the developed optical fibers were embedded in aluminum oxide matrix. A processing technique was developed to eliminate the debonding and mismatching problems. Several specimens of size 2.0 in x 1.0 in x 0.25 in were successfully fabricated, each containing one or two optical fibers embedded parallel to the specimen axis. Some of these specimens were tested with thermal cycling, and all of them were survived without damage or cracks. Brief descriptions of the

work carried out on each of the proposed tasks followed by results and discussions will be presented next.

II. 1. Optical Fibers Design and Fabrication

This task includes the study and fabrication of sapphire optical waveguides for high temperature applications and SEM examination of the developed fibers.

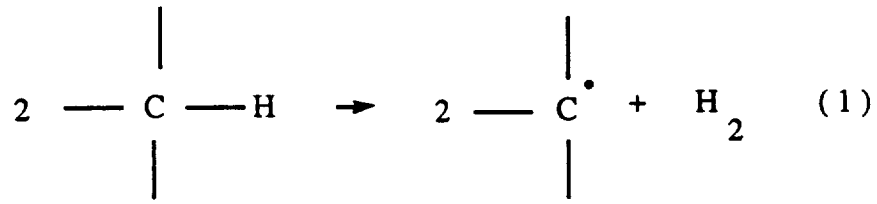
1. 1. General

A comprehensive study was performed on sapphire fiber waveguides (geometry, properties, and cladding and protective coating materials). The study also included a detailed investigation of different techniques for coating the waveguides, as well as fabrication. The coating material is important in the evaluation of the transfer of strain and temperature from the ceramic matrix materials to the sapphire fiber sensor element. In high temperature measurement applications, a coefficient of thermal expansion mismatch between the sapphire fiber element and the coating layers may lead to a loss of interfacial contact and result in discontinuity in heat flow and strain distribution at the boundaries. The quality of interface contacts and adhesion between successive layers that can be achieved during the coating process are very important; therefore, a surface treatment is required in order to promote a strong bond between the layers at the interfaces.

A novel chemical deposition technique developed by the project PI, Dr. M. El-Sherif et. al. of Drexel University, in collaboration with Dr. Don Roth of NASA LeRC, was applied and experimentally tested [12]. Optical grade single crystal sapphire fibers were used as the core of a multimode optical fiber. This sapphire fibers were coated with a thin layer of polycrystalline alumina by introducing very fine particles (20 nm) of the material in a polymerizable monomer carrier. The monomer is allowed to polymerize, leaving a uniform coating on the fiber surface. The polymer, polyacrylic acid, is compatible with the alumina fiber surface, giving good adhesion to both the powder particles and the fiber surface. Binder removal and subsequent sintering were performed by heating the coated fibers to 600°C and ~1400°C, respectively. This last process was optimized in order to eliminate porosity

and insure coating uniformity. This thin layer of fine particles provides the necessary bonds between the optical fiber core and the cladding material. For the cladding material's deposition, and to reduce the number of interfaces in the structure of the multilayer fiber, the same deposition technique was repeated to grown the cladding layer directly on the fiber core. In this way, the necessary requirements for strong bonds with the fiber core was achieved. The principles of the coating processing technique are presented next.

Monomers are linked to form polymers by various kind of initiations followed by propagation and, finally, termination. In polymerization, initiation of a polymer can be characterized as atomic or elemental. In this class of polymerization, chemical activators are used to promote polymerization at room temperature. Currently, most chemically polymerization's utilize an amino-peroxide initiating system (for example using the accelerator N, N-dimethyl-p-toluidine [DMPT] and the initiator benzoyl peroxide [BPO]). DMPT is added to the monomer (acrylic acid) and is interacts with the BPO. The product is added to the filler (fine particles of alumina or silica powder), to produce free radicals on the polymer side chains. These radicals produce C-C cross links by saturating one another. Thus, the cross linking in its simplest terms can be express as shown in the following figures:



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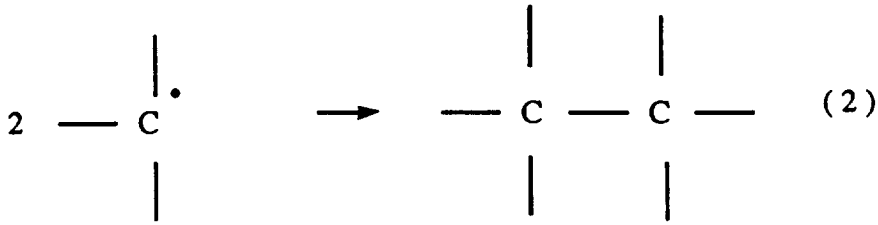

(2)

However, the cross linking mechanisms can sometimes be more complex. In various suspension systems, 0.05% to 2.0% of the accelerator DMPT and 0.5%

to 2.0% the initiator BPO are used for monomer polymerization, based on the weight of the monomer. The coating thickness can be controlled by varying the quantities of the initiator and the accelerator to adjust working time of the suspension before hardening. This technique was used for surface treatment and repeated for coating the cladding layer.

1. 2. Surface treatment and coating with the cladding layer

1. 2. 1. Preparation of homogeneous suspension

For this project, a suspension was prepared of 0.05 gm tertiary amine accelerator N-Ndimethyl-para-toullidine (DMPT) and 0.05 gm benzoyl preoxide (BPO) initiator added to 10 ml acrylic acid monomer and 10 ml H_2O. The hardening time required for this mixture is between one hour and half to two hours. Aluminum oxide (Al_2O_3) particles were added to the solution at different loading levels from 1 gm (10 wt %) to 4 gm (40 wt %) and the powder was mixed well to have a homogeneous solution. A spinner was used in order to promote homogeneity in the solution.

1. 2. 2. Coating the fiber with the cladding layer

There are several critical parameters involved in the coating process. Therefore, a large number of optical grade sapphire single crystal fibers were prepared and coated at different conditions. The diameter of these optical fibers were between 0.25 mm and 0.125 mm. These optical sapphire fibers were cleaned by hydrochloric acid (HCl concentration 36%) and washed in distilled water. Then each fiber was coated with a thin layer of fine particles to provide the necessary bonds between the optical fiber core and the cladding material.

Each fiber was immersed in a prepared suspension containing 10%, 15%, 20%, or 40% aluminum oxide, for half an hour, resulting in the deposition of a thin layer of the coating on the fiber surface. These fibers were left for 24 hours to polymerize in room temperature. The coated fibers were then heated to 600°C for binder removal. The time needed for complete polymer evaporation depends on the thickness of

the coating layer and the materials concentrations. Therefore, different times (10-60 minutes) were used in the interest of completeness.

The temperature was then increased for each fiber to a different degree between 1300°C and 1500°C for varying time duration's (10-60 minutes), in order to sinter the alumina powder around the fiber. The heating and cooling rates for the furnace were in the 2-10°C/min range. The same procedure was repeated with different mixture concentrations for coating the (20-30 μm) cladding layer. The thickness of the cladding layer was controlled by changing the deposition rate (changing the concentration of the materials in the mixture and/or changing the deposition time).

1. 3. Coating the fiber with a protective layer

Thin film coating of silicon carbide over the fiber cladding layer was performed using the plasma enhanced chemical vapor deposition (PECVD) technique. An ion-assisted PECVD process was used for coating the optical fibers at room temperature. The substrate was placed on the powered electrode in a capacity coupled, asymmetric parallel plate reactor powered by 13.56 MHz rf. The feed gas consisted of a mixture of tetramethyl silane and hydrogen. The substrate holder was maintained at a constant temperature by a recirculating cooler. The pressure of the reactor was maintained below 200 torr. Due to the asymmetry of the reactor, a high self bias voltage was developed on the powered electrode. This enabled high energy bombardment of ions on the substrate and makes the deposition possible at low temperature. These reactor parameters are varied to obtain films with desired properties. Adjustable deposition parameters include chamber pressure, gas ratio, power level, and substrate temperature. These parameters were adjusted for deposition of 0.25 μm silicon carbide layer on the top of the cladding layer.

1. 4. SEM examination of the developed sapphire optical waveguide

Based on the work carried out and explained before in sections II.1.1.-1.4., several samples of optical grade sapphire fibers were coated with the cladding and protective layers, as discussed before. At each stage of the coating

process, the surface morphology of the coated fibers were examined and studied using a scanning electron microscope (SEM). At first, the prepared optical grade sapphire fibers were examined by SEM before coating. The results show a very smooth surface with negligible variation in the fiber diameter. Then the samples were studied after coating with different suspension concentrations. It was clear that the coated layers were not homogeneous and/or the sintering process was not completed in the majority of these samples. For these samples, the alumina concentration in the solution might have been low, or the heating temperature or time may not have been sufficient. Another major observation made was that diffusion started at 1500°C. At this temperature, the surface morphology of the fiber started to change at 1500°C. Accordingly, the maximum temperature used for our experimental investigation was limited to 1400°C.

Regardless of this limitation, the results show that a few of these samples were perfectly coated. It is encouraging, however, to find that the samples with 20% concentration of alumina produced the highest level of homogeneous thin film coating required for surface treatment. Several additional experiments with this coating concentration were prepared, and the results were consistent. For these samples, the fibers were heated to 600°C for one hour to ensure that the polymer was completely removed, and the temperature was then raised to 1400° C for an hour for perfect sintering of the alumina powder on the surface of these fibers. A thin layer of homogeneous alumina is coated on the surface of the optical fiber. Finally, in order to coat the cladding layer to a thickness of about 20 μm, higher alumina concentration was used. The fibers were coated with three layers of the prepared suspension in sequence before they were heated. The thickness of the cladding layer was controlled by changing the concentration of the materials in the suspension. In this manner, a high quality cladding layer was successfully obtained. At each stage of the coating process, several samples were examined and studied using a scanning electron microscope (SEM).

During the course of this task, several difficulties arose in determining key parameters required to optimize this process. Such difficulties include:

a. How can a homogenous suspension containing alumina or silica powder be prepared ?

b. What are optimal concentrations of initiator and accelerator in suspension to change conditions for polymerization ?

c. What is the time needed to grow a uniform layer of the polymer/alumina mixture on the surface of the optical fiber ?

d. What are the time and temperatures required for heating the fibers to remove the polymer completely and to sinter the alumina particles in the cladding layer?

All of these questions and others were addressed during our investigation to optimize the coating process.

Then, the fibers were coated with the protective layer. A thin film coating of silicon carbide over the fiber cladding layer was performed using the plasma enhanced chemical vapor deposition (PECVD) technique. The pressure of the reactor was maintained below 200 mtorr, and the parallel plate electrodes were connected to a 13.56 MHz rf supply. This enable high energy bombardment of ions on the substrate and makes the deposition possible at low temperature. These reactor parameters are varied to obtain films with desired properties. Adjustable deposition parameters include chamber pressure, gas ratio, power level, and substrate temperature. These parameters were adjusted for deposition of 0.25 μm silicon carbide layer on the top of the cladding layer. The PECVD technique was used to provide a uniform thin layer of SiC on the top of the cladding layer.

This task has been completed in the first 6 months of the program (last year), and the results have been presented in the semiannual report submitted October, 1992. In addition, brief summary of the performed work is recorded in Ref.[9].

II. 2. Processing of Ceramics Containing Sapphire Fibers

2. 1. General

The key issue of embedding optical fibers into ceramic specimens is to avoid any defects such as cracking and debonding during the process. Although the fibers are covered with a protective layer of coating compatible with the surrounding ceramic material, cracking may takes place during the sample making process. Also, cracks may appear within the ceramic base material itself, and not at the interface of fiber and ceramic matrix.

Therefore, this task was focused on the development of a processing methodology to incorporate sapphire fibers in ceramic matrix materials. Several approaches for processing of the smart ceramic samples were tried. Several difficulties arose in the impregnation process, however, a promising technique has been developed and tested. Several samples of 2 in x 1 in x 0.25 in aluminum oxide coupons with one or two embedded sapphire fibers were fabricated and successfully tested with thermal cycling.

Currently, there is no quick and easy way to produce a ceramic matrix having an optical fiber embedded with exposed leads, as protecting the leads of the optical fiber during the impregnation process is a major concern. In preliminary tests, a few samples were produced using conventional manual green body methods. However, they had flaws and cracks upon drying due to the lower density of the green bodies. If the specimen is compressed in a mold specially designed to remove the excess water, a dense body could be made. This mold would allow the optical fibers to be embedded during the forming process, and because of the even pressure exerted during compression, major flaws in the material would be avoided. Therefore, the designed mold would include a stainless steel filter to release excess water. Therefore, the developed process of making ceramic samples with embedded fibers is based on the pressure filtration method [13], which is illustrated in Fig. 2. The method involves filtration of a fluid from a colloidal suspension, and at the same time the compaction of a particulate network. The advantage of using this method is the uniformity of the particle distribution. As shown in the figure, the process include steps of powder mixing and stirring, pressurized casting, and sintering. Several samples were

successfully made using the pressure filtration method. A brief description of the processing method is presented herein:

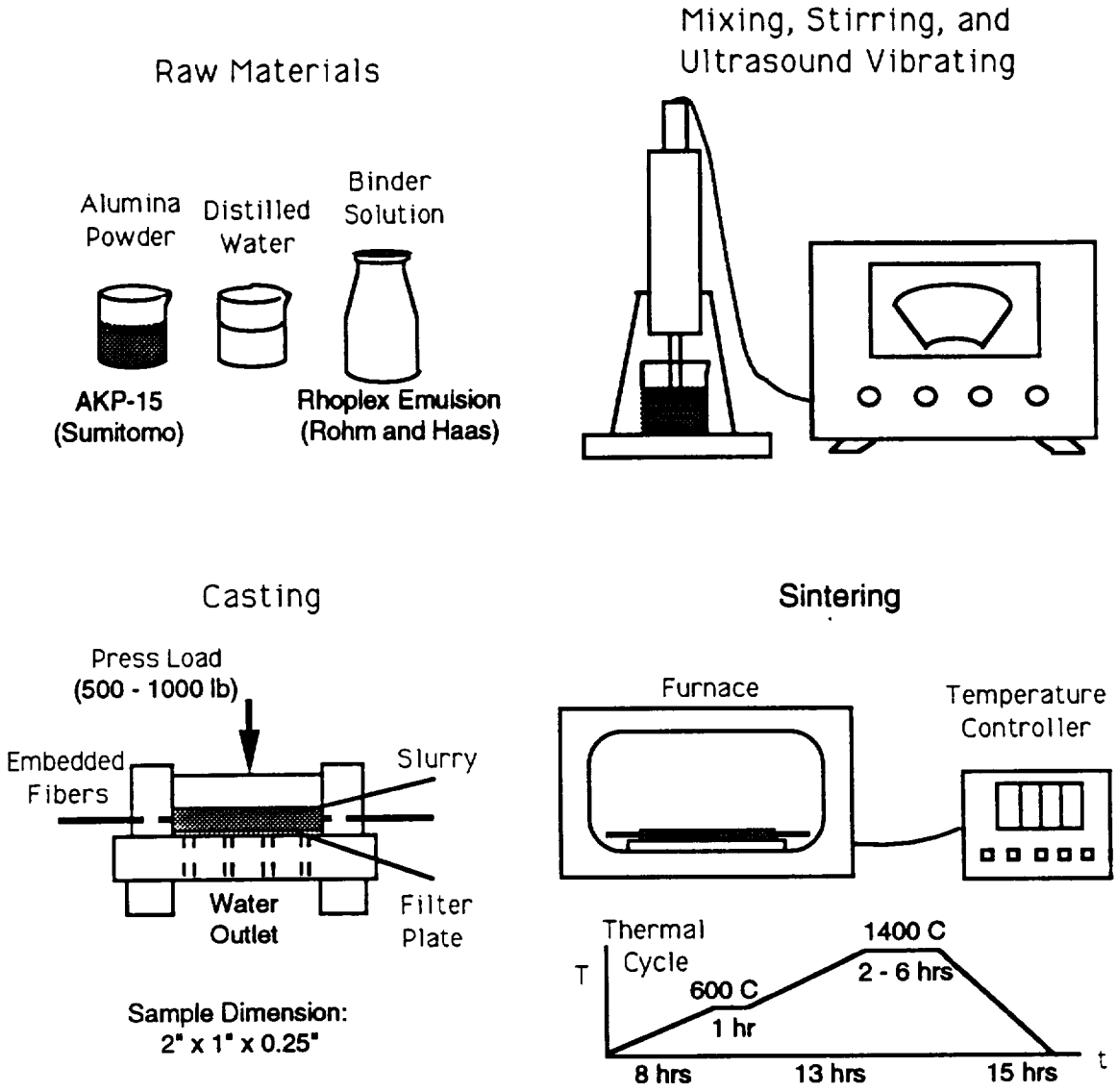

Fig. 2 : Processing of ceramic sample containing optical fiber.

2. 2. Powder preparation

Sample preparation starts with raw materials in powder form. AKP-15 Alumina powder from Sumitomo Chemical Co., New York, NY, was used as the base material. The alumina powders were first mixed with distilled water at a volume ratio of about 1 to 4. The mixture was stirred continuously to break up powder agglomerates in the suspension. An ultrasonic vibrator was also used to assist the powder-breaking process. When the suspension

appeared as a good mixture, a binder solvent (Rhoplex aqueous acrylic emulsion from Rohm and Hass, Philadelphia, PA) was added to the slurry system. For an alumina powder mass of about 17.0 grams, 2 ml of the binder solution was added. Stirring was continuously applied while the binder was being dropped into the slurry mixture. Stirring usually lasts about fifteen to twenty minutes until the slurry becomes a good viscous mixture. An ultrasound vibration probe was then put into the mixture to perform the vibration for about ten minutes. The vibration breaks the powder into much tiny particles mixed further with the fluids. The slurry was then ready for the casting operation. Similar sample preparation and material selection process is reported in Ref. 14 for making ceramic sheet samples.

2. 3. Pressurized casting and sintering the materials

In the casting process, slurry was poured into a mold with a filter plate and a porous base plate at the bottom as the water outlet. Fibers were pre-placed into the mold cavity through pre-drilled holes on the side plates of the mold, with both ends extended out of the mold. Slurry was pushed into the mold corners and around inserted fibers manually. Then a top plate was placed onto the slurry. Compressive load was applied through the top of the mold via a press. While load was gradually increased, water was squeezed out of the slurry and the alumina powder was compacted. Press load was gradually increased to 500 to 1000 l, depending on the sample, for 30 to 40 minutes, and then was held at maximum load for 10 to 15 minutes. After that, loading was decreased gradually at about the same rate. After the load cycle was finished, the sample was taken out of the mold and placed onto a porous base to be dried for at least 24 hours via natural convection.

In the sintering process, the sample was placed into a furnace. Temperature was gradually increased to about 600^{0} C at a rate of about 1^{0} C per minute, and kept for about one hour. Debinding takes place at this temperature. Then temperature was increased to about 1400^{0} C at the same rate and kept for a few hours (usually between 2 to 6 hours) for sintering. After the sintering, the sample was cooled down slowly to room temperature. Several coupons were successfully fabricated and preliminary tested for thermal cycling. The results will be discussed next.

2.4. Results on processing of ceramic samples containing optical fibers

The main advantage of the pressure filtration method is that a uniform high density matrix can be achieved, therefore, the cracking sources can be minimized. A pressure filtration cell was designed for this application. This pressure-filtration cell consists of a stainless steel filter surrounded by four walls of acrylic plastic, as shown in Fig. 3. The filter is supported by a plastic base with holes drilled through it for the water outlet. This structure was designed to facilitate removing the sample from the mold without breaking the leads of the optical fibers. Four c-clamps are used to hold the walls of the cell tightly. Pressing of the plunger is a critical issue and must be performed slowly to prevent bending of the fibers. Also, the shear forces between the wall and the slurry have to be monitored closely in order to prevent fiber breaking.

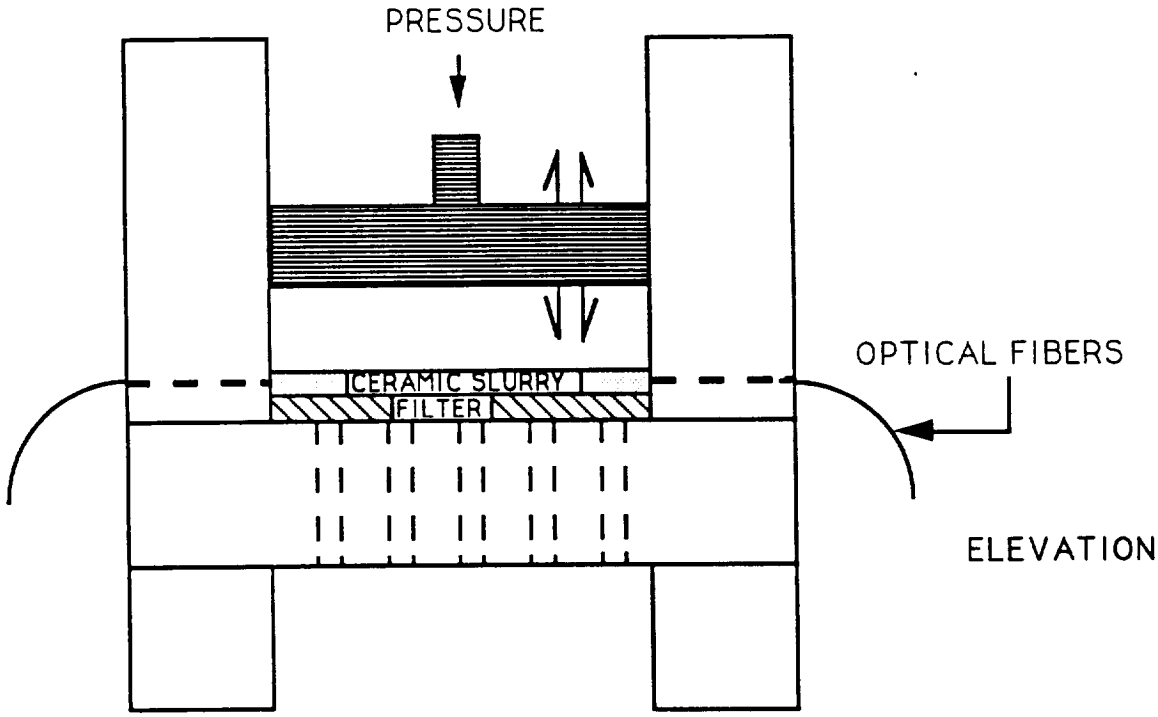

Fig. 3: The pressure-filtration cell using in processing ceramic composites containing optical fiber.

The main parameters involved in the process are the status of the slurry mixture or viscosity, the magnitude of the press load, the loading rate, and the thermal cycle during the sintering.

Viscosity of the slurry is an important parameter to the casting process. If viscosity is too low, powder is to be squeezed out with water during the pressing. On the other hand, if viscosity is too high, the flow of the material is difficult. Voids may be formed during the pressing. Currently, the viscosity or status of the slurry is visually checked. The viscosity can be changed with a different powder/water ratio. The current ratio of these elements is determined after a few preliminary trials, and was found to be in the right range.

Press loading is directly related to final ceramic sample density. However, with optical fibers embedded into the ceramic sample, the applied press load tends to bend fibers during the pressing. Therefore, this step is a critical issue and must be performed carefully in order to prevent bending of the optical fiber. If the load carried by the fibers is too high fiber breakage may occur. In the current casting setup, the press load range was found to be in the range of 500 to 1000 lb. on a 2 inch by 1 inch sample area. Fibers were slightly bent after the pressing, but can be kept from breakage.

Sintering involves mass flow and diffusion of the granule materials, which is a time-dependent process. Heating and cooling rates play an important role in the stress status of the fiber/ceramic samples. The sintering thermal cycle also induces thermal stress within the material. These stresses, if not properly controlled, cause material deformation resulting in excessive bending or distortion, and even cracking. Slow heating and cooling rates can help to reduce the stress within the material. Longer holding time at the sintering temperature also provide more uniform mass diffusion and granule distribution.

Experiments on embedding optical fibers into ceramic samples, performed during the first year of the project, provide valuable information about the process parameters. Several ceramic samples with embedded fibers have been successfully made using the pressure filtration method. These samples were preliminarly

examined by thermal cycle testing. Three samples were heated to 1400°C for three hours several times (four times). The heating and cooling rates for the furnace were in the 2-10°C/min range. All of the samples survived without damage or crack. More thermal cycling tests are in progress and will be continued for the next few months.

II.3. Development of The Experimental Set-Up and Optical Testing

3.1. The experimental set-up and the computerized data acquisition system

The use of optical fibers as stress sensors is dependent on changes in the geometry and index of refraction of the optical fiber by external perturbation. Applying mechanical or thermal stresses to a composite structure containing an embedded optical fiber will induce strain in the fiber. This induced strain changes the boundary conditions of the core and causes a redistribution of the launched optical modal power. This redistribution can be measured and then related to the induced perturbations.

Current research involving the use of optical fibers as internal sensors relates perturbations to changes in total intensity of the light signal transmitted through the optical fiber. Other methods involve launching coherent polarized light through an embedded optical fiber and observing the changes in the polarization or phase of the launched signal in the presence of external perturbation. The first technique (intensity modulation) is not sensitive enough for most applications, and the others (phase or polarization modulation) are only applicable to single mode fibers. The difficulties of producing single-mode sapphire fibers limit the application of the phase or polarization modulation techniques to these fibers.

For this project, a novel method is applied. This method depends on the analysis of the spatial distribution of the modal power in a multimode fiber. This distribution known as the Modal Power Distribution (MPD), and can be related to the external perturbation. The MPD technique has been shown to be much more sensitive than current methods applied to multimode fibers,

which merely relate applied stress to the total intensity of the optical signal. The testing system specifically designed for the detection of the modal power, coupled with computerized data acquisition system, is shown in Fig. 4. It consists of;

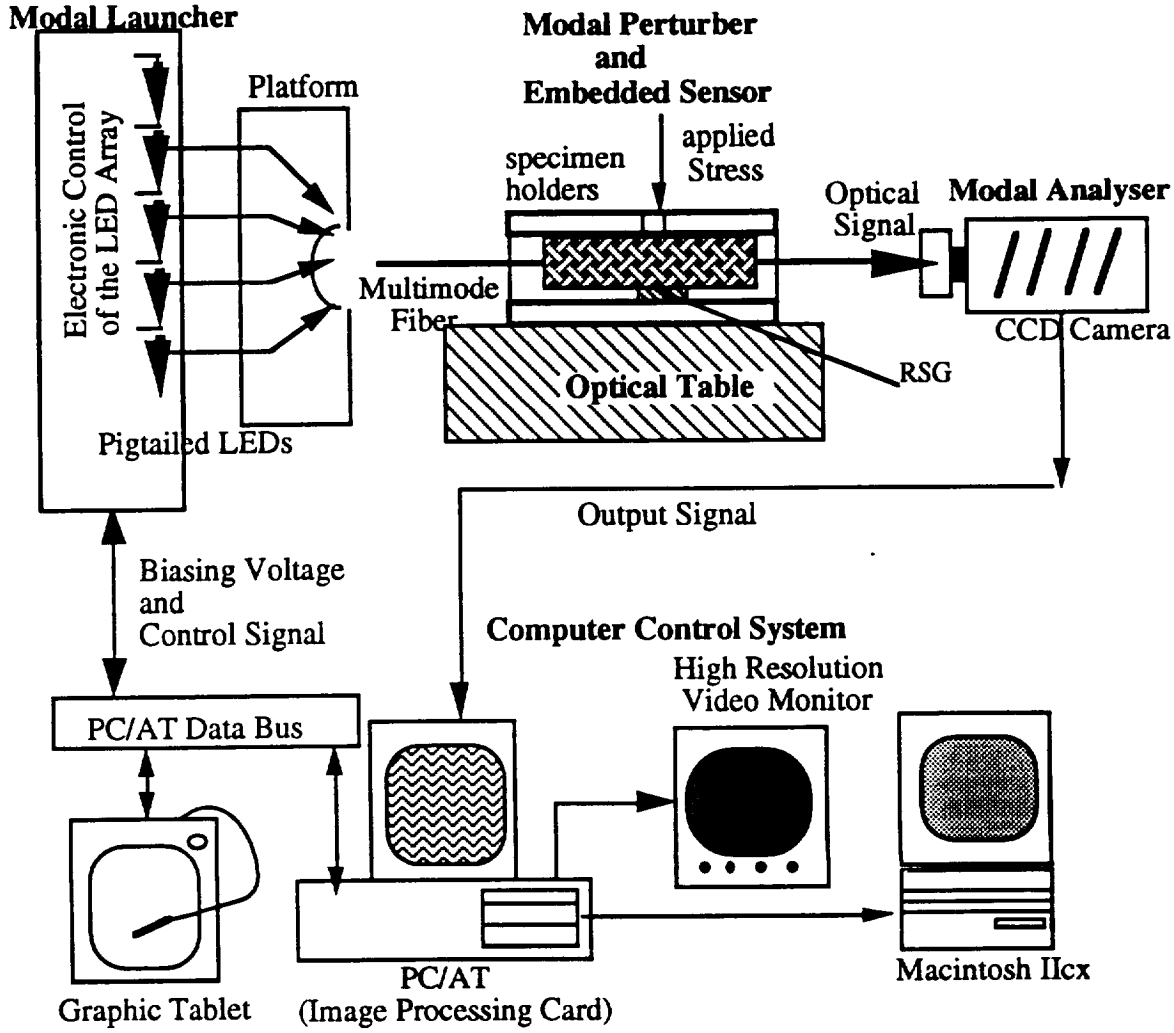

Fig. 4. Schematic diagram of the experimental set-up for testing sapphire fibers and smart structures.

i. The Modal launcher, which is designed to launch particular modal structures into the optical fiber. It has the sole objectives of exciting the specified modal groups in the fiber, using an array of AlGaAs LEDs.

ii. A high-sensitivity, high-resolution charged-coupled device (CCD camera), which is used as a modal analyzer to provide a live image of the far-field pattern at the fiber output.

iii. The modal perturber, which is used to apply quantified stresses in different ways to optical fiber or the embedded sensor of the smart structure. A resistive strain gauge (RSG) device is employed to control and calibrate the quantifiable applied stresses.

iv. Software to access the image processing system. With this software, it is possible to scan new images, plot the profile (vertical or horizontal) from frozen pictures, and save information.

v. PC/AT to control the whole system via data bus. It consists of; Image Processing Card, Math Co-processor (80287), RAM (1Mb), Processor (80286), Hard Disk, and Floppy disk.

vi. High resolution video monitor working in the Analog System. This device provides the life image of the optical fiber far-field pattern.

vii. Macintosh IIcx to edit graph data. The profile data can be transferred from the PC/AT to Macintosh via cable using Versaterm software. The data can be saved in Cricket Graphic and edited to obtain the graphs presented in the measured data.

Recently, a short study has been performed in collaboration with the Wright-Patterson Air Force Base for testing the application of the MPD technique to sapphire fibers. A signal processing and analysis program using a combination of several 3-D imaging algorithms was developed. Opto-mechanical testing of sapphire fibers was successfully performed. The results show that the developed program is compatible with the MPD technique and capable of providing a detailed structure of the optical fiber far-field pattern in 3-D [10]. In this project (with NASA LeRC), the developed program will be enhanced and employed for optical testing of sapphire fibers and to establish the correlation functions. The program provides Mac Paint-like editing of color and grayscale images. It can flip, rotate, invert and scale selections. It supports data translation and

scion frame grabber cards for capturing images or movie sequences using a CCD camera. These advantages can explore the potential of the MPD technique for applications with sapphire fibers. Experimental investigation was performed to prove the concept of using sapphire fibers for sensor application. The experimental set-up shown in Fig. 4 was used to excite a limited group of modes of the fiber under test and to scan the far-field pattern.

Due to the crystallographic structure of the fiber, which is different from amorphous silica fibers, the patterns of the excited modes were broader than that obtained before with amorphous silica fibers, under the same conditions. This is due to diffraction and interference of optical signals by the crystal planes. Special algorithms were used to filter interference noise and limit its effect. Regardless of the crystal effect, we were able to apply the MPD technique to the fiber and successfully detect changes in the far-field pattern induced by external stresses. Stresses were applied in the form of transverse compression loads. Certainly, the developed signal processing algorithms were used to limit the crystallographic structural effect and to enhanced the far-field pattern. In addition, these algorithms were used to dramatically improve the signal to noise ratios, which will enhance the sensitivity of the sensor. Samples of the results are presented next.

3. 2. Results on the application of the MPD technique

Several sapphire fibers were tested under various stresses, using the experimental set-up shown in Fig. 4. The light launching angles used during most of the experimental investigations were between 10 and 7.5 degrees. These launching conditions were used to excite higher order modes, which are more sensitive to external perturbations than lower order modes. The optical fibers were positioned on the testing optical table in such a way as to facilitate the application of external stresses. The external perturbations were applied in the form of a transverse compression load. The stress imparted to the optical fiber was measured by a resistive strain gauge (RSG) positioned under the fiber.

The effect of external perturbations on modal power distribution (MPD) was predicted by scanning the far-field pattern. At the output end of the fiber, the far-field pattern emerging from the fiber was scanned by a high-sensitivity and high-resolution CCD camera. The camera output was monitored through an image processing board installed in the computer controller. The board contains a frame grabber and digitizer enabling freezing of the line video and access to the gray level of each pixel. Special algorithms were used to filter the interference noise and improve signal to noise ratios. Several measurements were taken while adding (increasing) the stress on the fiber, and during relieving (releasing) the stress. The preliminary results show that there is not much difference in the results obtained during adding stresses or relieving the same stresses. Therefore, for the feasibility study, due to the size limitation of the computer memory, only the data obtained during the process of increasing the applied stress have been saved and processed. A 3-D image processing algorithms were used to enhance, analyze, edit, and display the far-field pattern. Also, they were used to predict the far-field pattern profile (the MPDs) for each scan.

Samples of the final results obtained for an optical grade c-axis sapphire fiber prepared by the Edge Fed Growth Puller technique are shown in Figs. 5-11. The first figure (Fig. 5) shows the 3-D far-field pattern recorded at the fiber output, before any stress was applied, i.e. the MPD with no applied stress (initial state). The fiber was excited with an optical beam at an angle 8^0 off-axis. The normalized output intensity profile of the far-field pattern vs. the scanning angle is also shown on the same figure. It is clear from this figure that the crystallographic structure of the fiber has resulted in a radial periodic interference pattern. However, the effect of the interference noise was reduced by using a special image processing algorithms. Also, this figure shows that optical intensity for the lowest order modes (on-axis modes) is almost zero, and most of the power is concentrated at an angle equal to the excitation angle.

In the presence of a weak load (0.212 kg) applied to the optical fiber as transverse compression load, an induced change in the MPD was detected, as shown in Fig. 6. This figure shows that the sensitivity of the sensor is very high. As the applied load was increased (from 0.212 kg to 0.556 and 0.803

kg), considerable rearrangement of the modal power was shown. This can be seen in Figs. 7 and 8. Each of these stresses causes an intermodal coupling leading to a modal power redistribution, which is broadened toward smaller angles reflecting a transfer of power to lower-order modes. For higher stresses, more power transferred to the lower order modes, as shown in Fig. 9 corresponding to 1.152 kg. Increasing the applied, more, the MPD becomes nearly flat at 0.0^{o}, as shown for load levels 2.285 and 2.951 kg, (Figs. 10 and 11). However, the change in the total output power at all of these stress levels was still negligible. Further increase of applied stress (over 3.0 kg) results in a small optical loss and decrease of the total throughput. This indicates that measurements on modal power distribution are much more sensitive than total intensity modulation for this type of application. Based on the results shown in Figs. 5-11, a simple relation was drawn for the on-axis intensity of the far-field pattern in response to the applied loads, as shown in Fig. 12. This figure shows a uniform smooth variation in optical intensity at the center of the fiber induced by changes in the applied stress. As a result of this simple correlation, a direct approach and cost effective methodology is suggested. In this approach, the CCD camera can be replaced by a single photodetector. This detector will be positioned on the fiber axis to collect only the power of the lower order modes. The output of this detector can be coupled to a simple biasing circuit to indicate the change % in the optical intensity output, at the center of the far-field pattern. An additional photodetector, positioned at a different angle from the fiber axis, can be used to provide a reference signal.

From experimental observations, signal intensity changes as external load varies in magnitude. The correlation function between signal intensity and applied stress has not been fully developed yet. During future work, theoretical models of the optical MPD will be compared with measured 3-D far-field patterns. Examples of the 3-D image, recorded for the far-field pattern are shown in Figs. 13 and 14 for the stressed and unstressed fiber presented in Fig. 11 and 5, respectively. These 3-D measurements will be used during the Phase II program to verify the theoretical model. Based on these results the application of the MPD has been shown to be a sensitive, simple, and cost effective tool for sapphire fibers applications.

III. Novel Concepts

A suitable technique for homogenous coating of sapphire fiber has been developed. This coating technique is crucial for the fabrication of reliable optical waveguides for a broad range of fiber-optic sensors. Preliminary investigations with this approach appear to be very promising in terms of confining optical signals within the fiber core and providing an inexpensive and highly sensitive optical sensor. The combination of the spatial modulation technique with multimode sapphire fibers appears to be an attractive methodology for the development of optical sensors for very high temperature applications. A Patent Disclosure is in process through Drexel University in collaboration with NASA LeRC regarding the developed process,. A copy of the disclosure is shown in Appendix A.

IV. Future Plan

The work plan for this quarter (July, August, and September) will be focused on testing the optical response of sapphire fibers and on performing the thermal cycling tests. This will includes fabrication of a number of coupons with embedded fibers. However, due to time limitations imposed by sharing the only high temperature furnace available to the Department of Materials Engineering, we would like to modify the proposed thermal cycling tests without changing the technical objective or the proposed costs. Based on the total number of hours this furnace was used last year, several difficulties arose in fixing it and providing required maintenance; to insure running all of these tests in the next few months, we are requesting minor modifications to the thermal cycling tests as follows:

i. Since we are going to run these tests (series A and B) at 900°C and 1300°C, the tests at 1100°C can be eliminated.

ii. Series B can be modified to 10 and 50 cycles at 900°C and 10 and 20 cycles at 1300°C.

We appreciate your understanding of the problem and your approval on these modifications.

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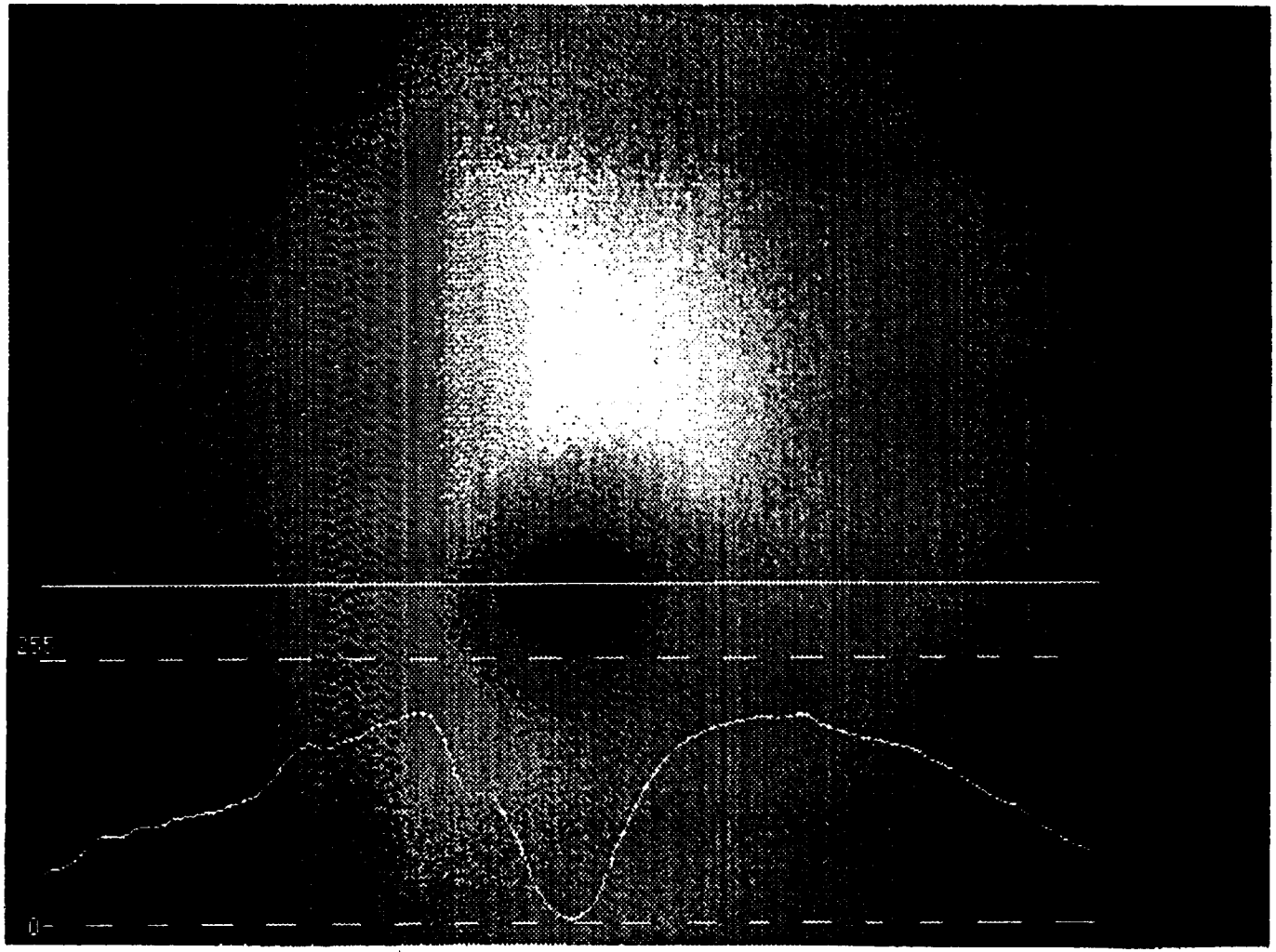


Fig.5. Indicates the far-field pattern and the horizontal intensity profile under no stresses.

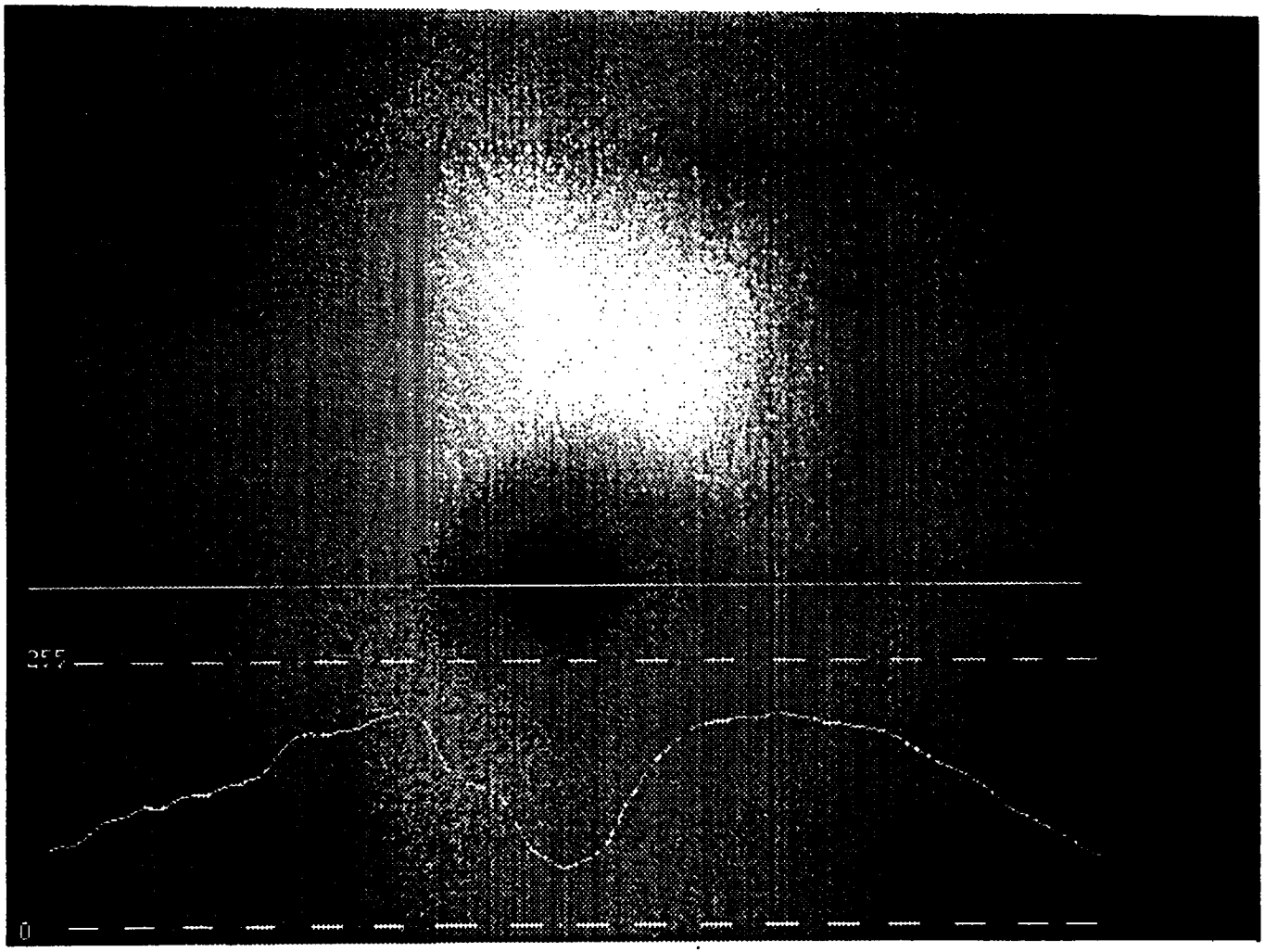


Fig. 6. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 0.212 kg applied at one location on the fiber surface.

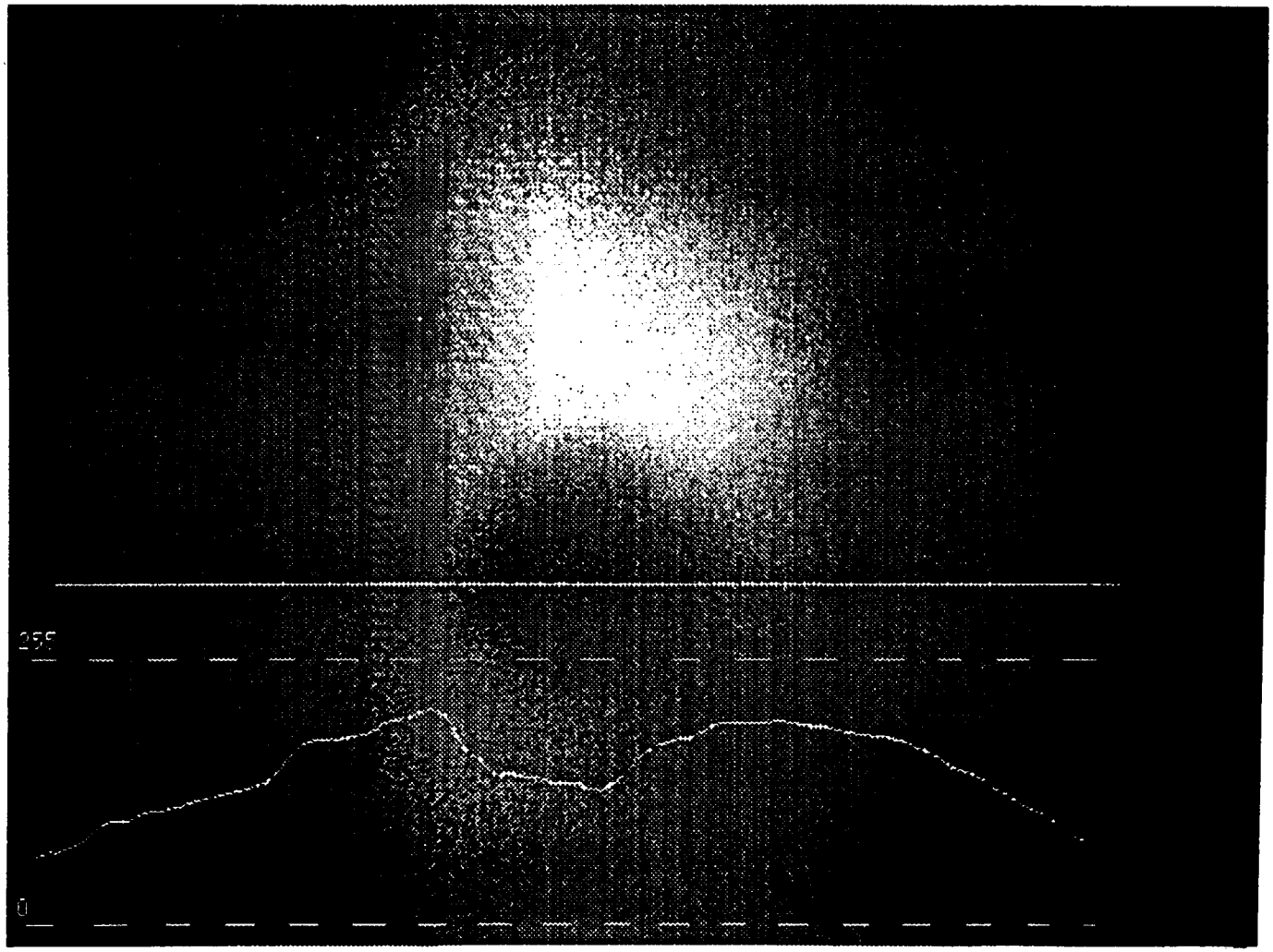


Fig.7. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 0.556 kg applied at one location on the fiber surface.

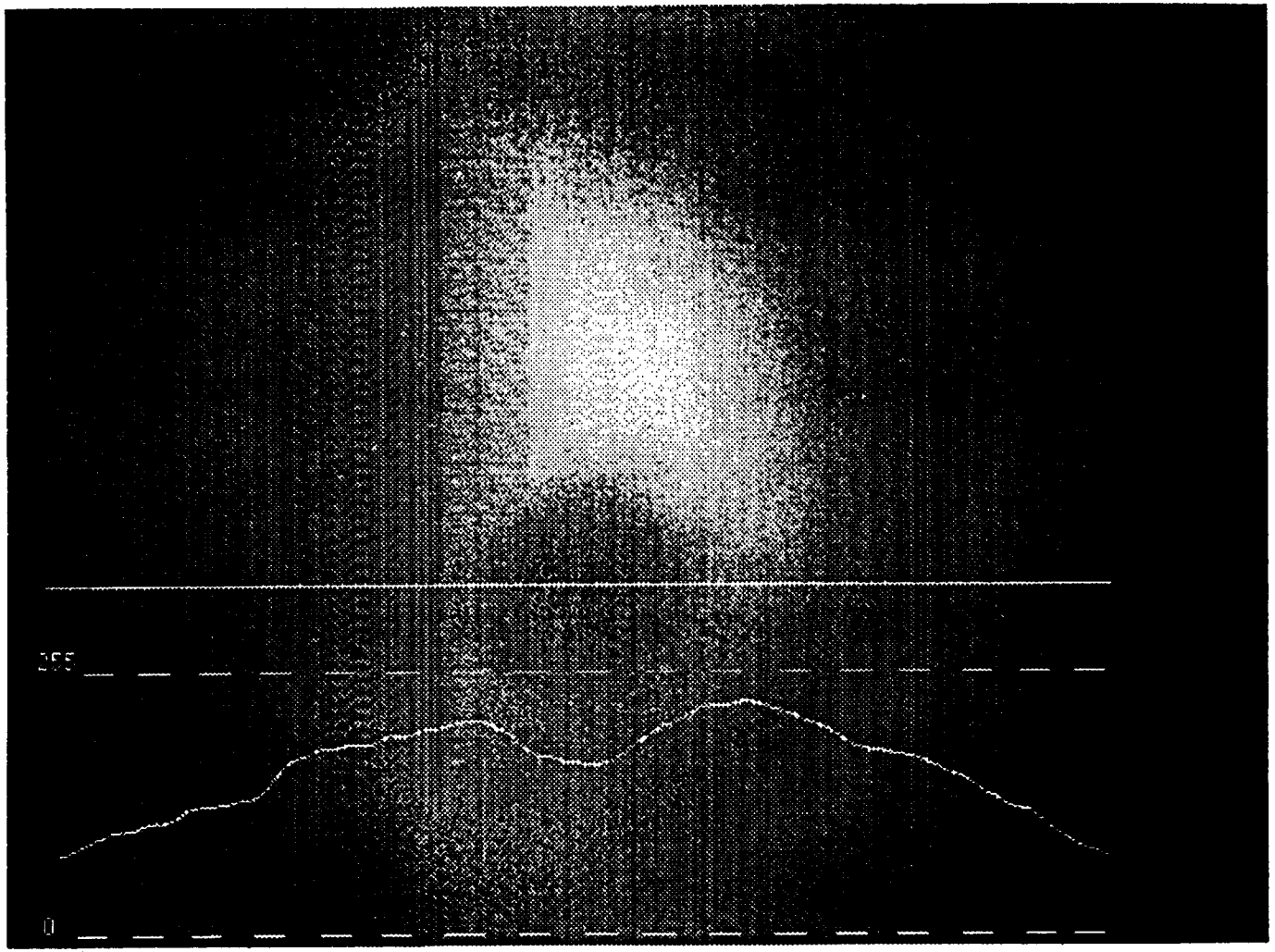


Fig.8. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 0.803 kg applied at one location on the fiber surface.

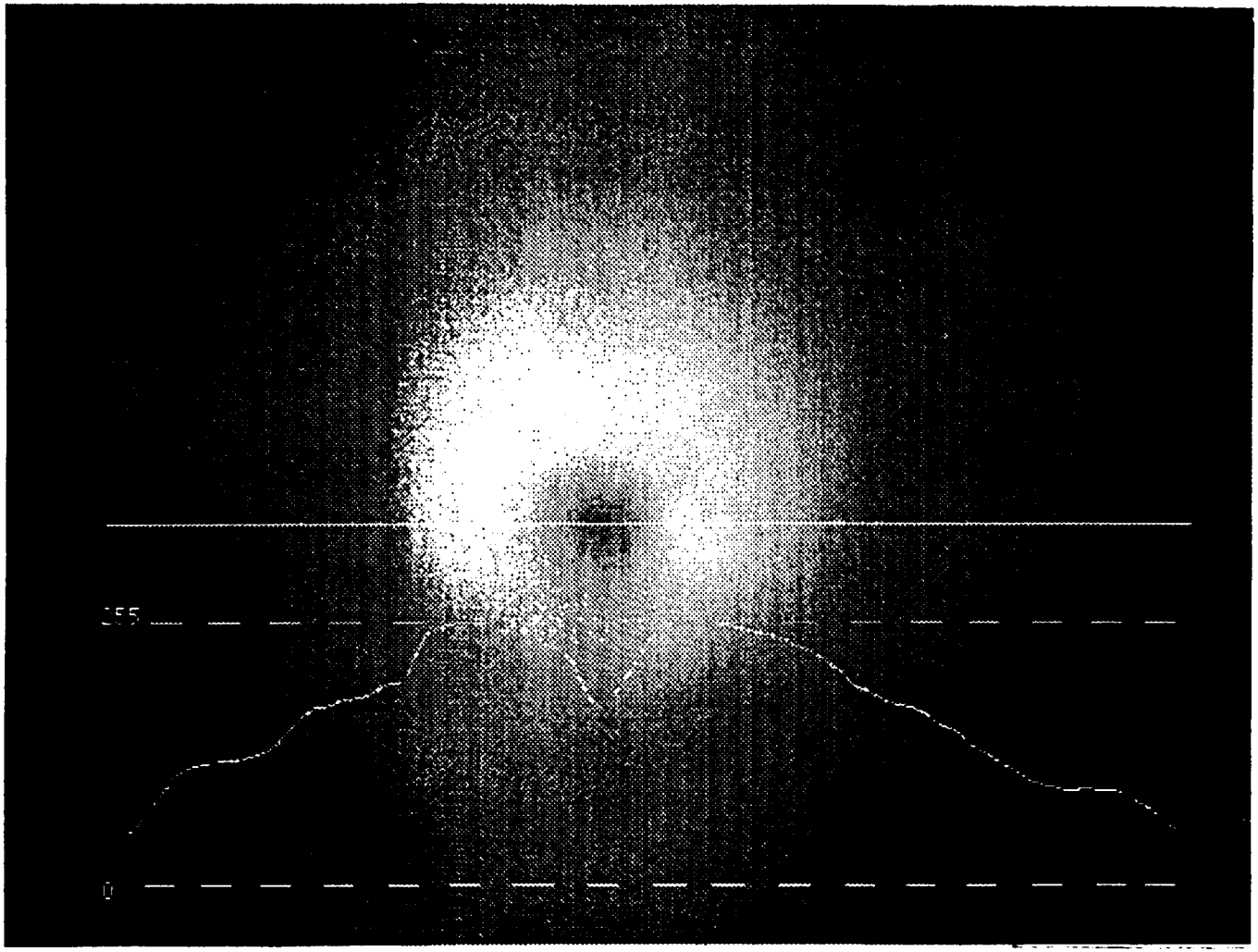


Fig.9. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 1.152 kg applied at one location on the fiber surface.

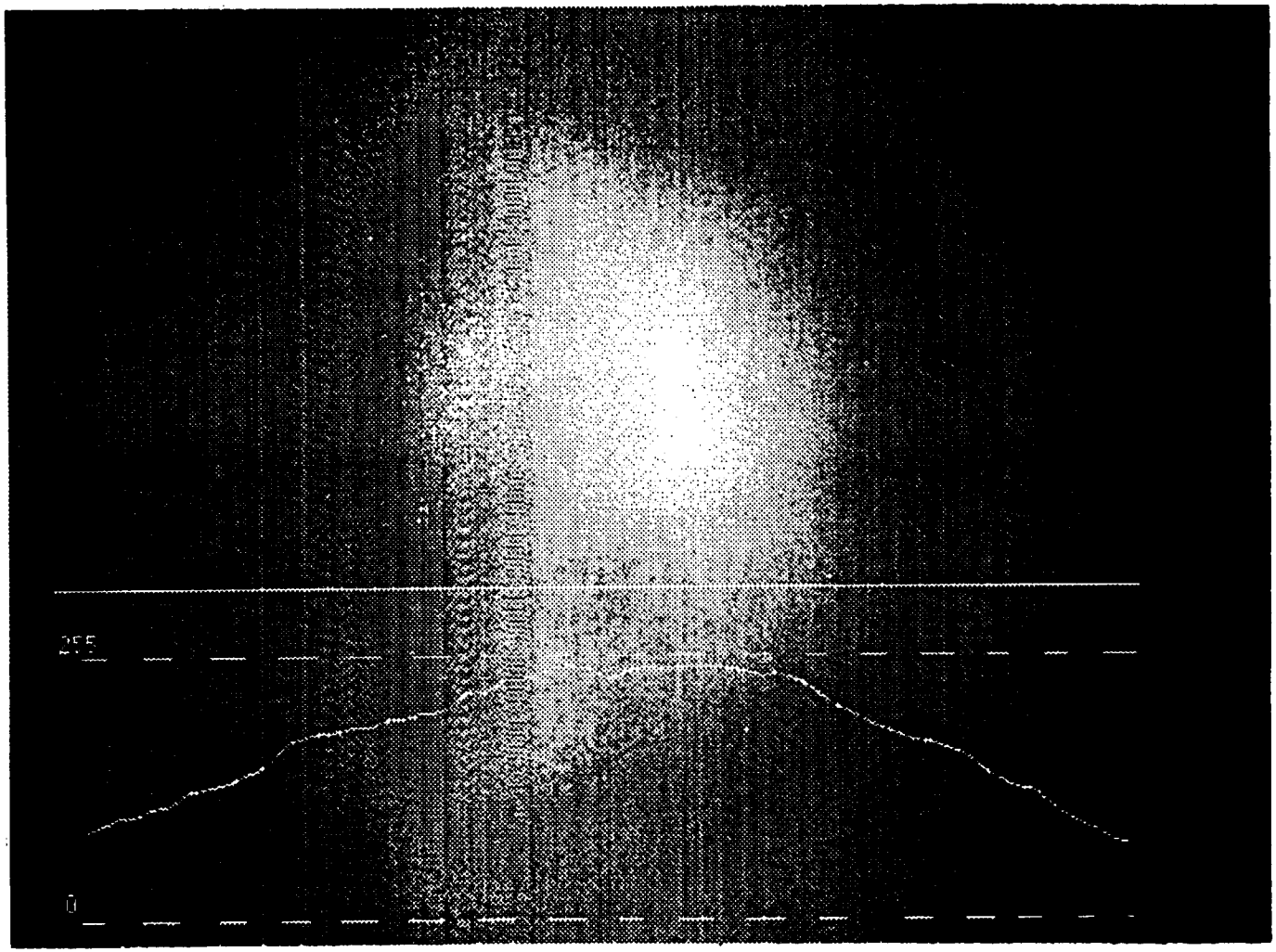


Fig.10. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 2.951 kg applied at one location on the fiber surface.

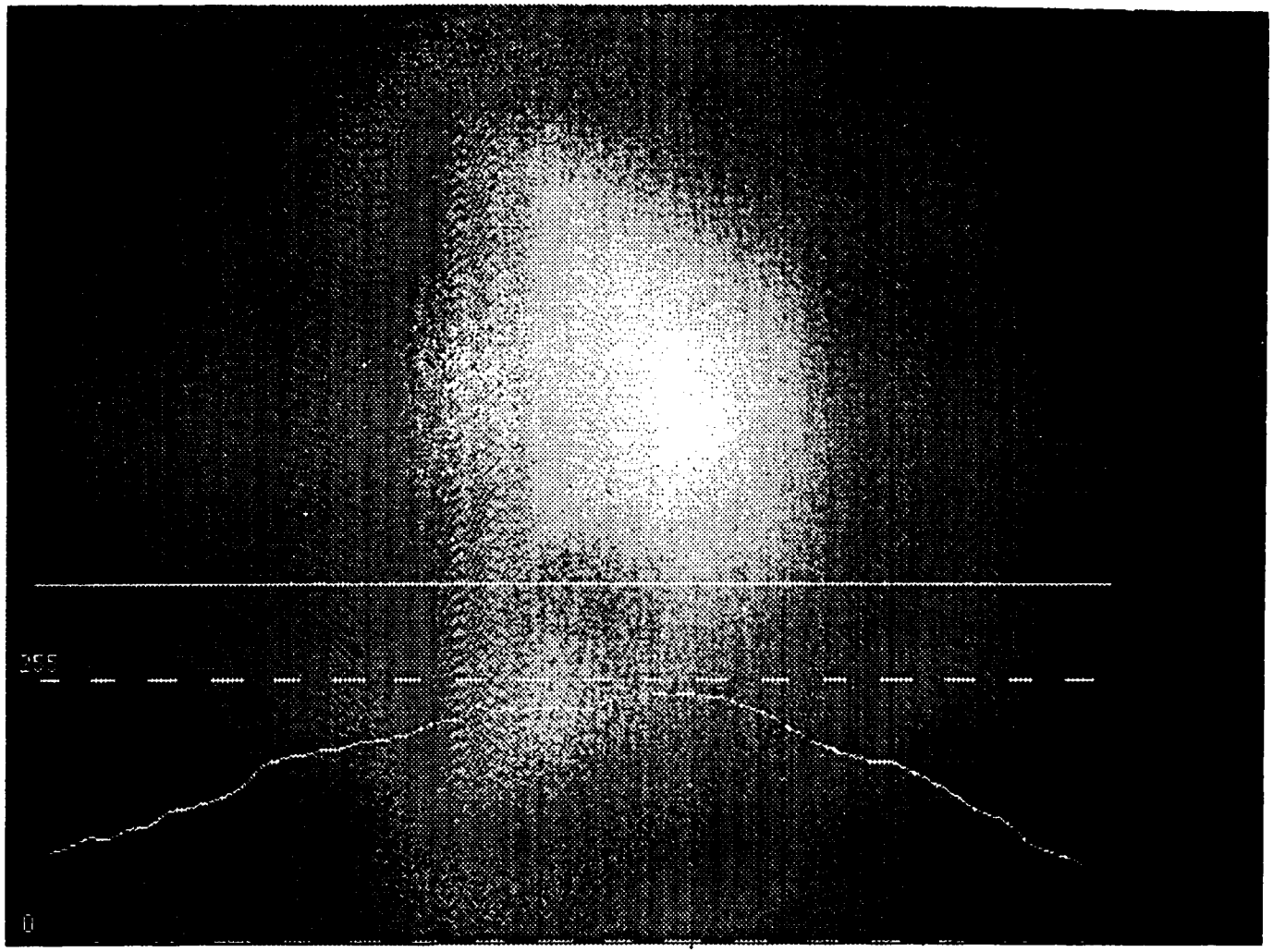


Fig.11. Indicates the far-field pattern and the horizontal intensity profile under transverse compression stress of 2.285 kg applied at one location on the fiber surface.

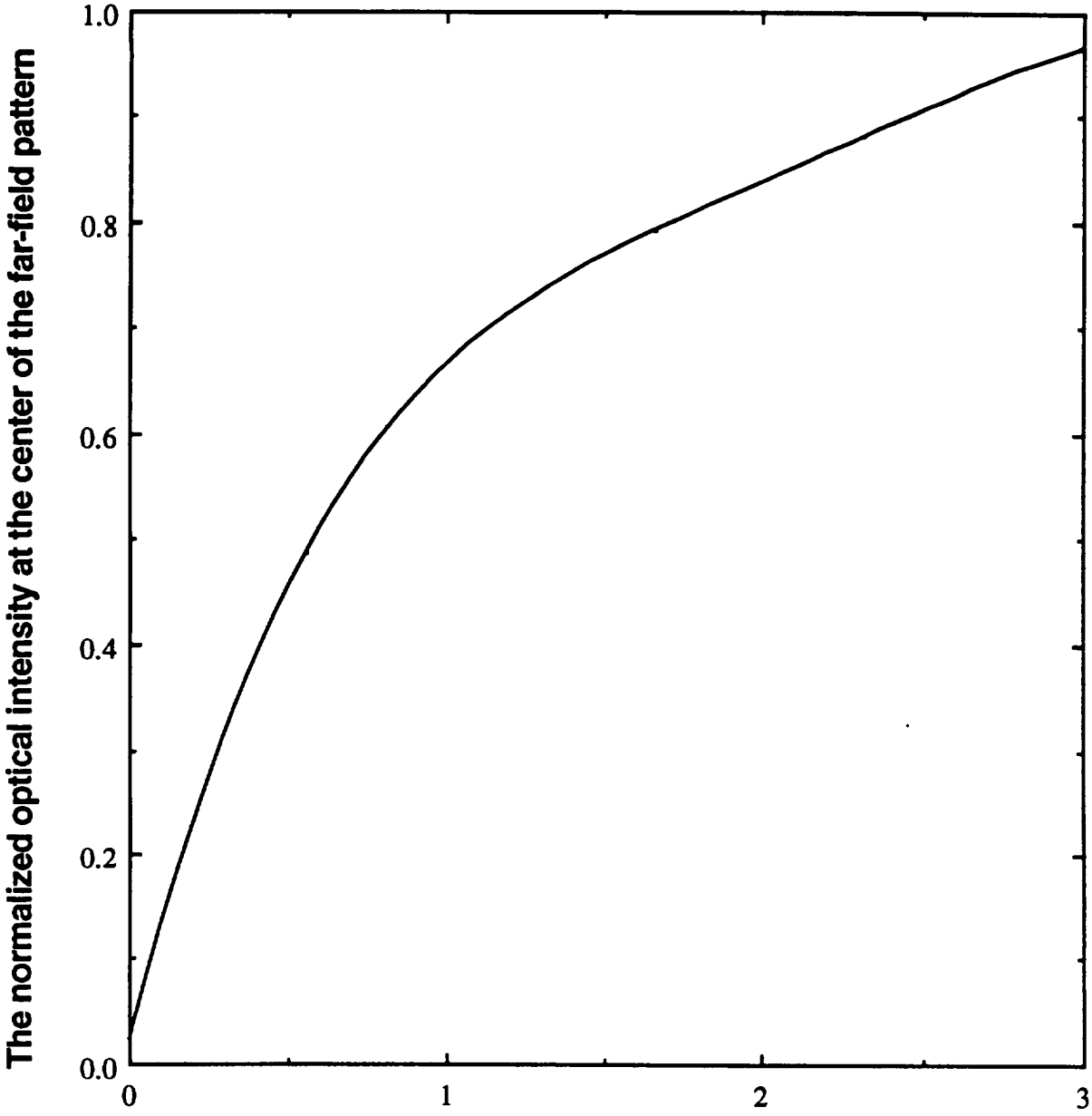


Fig.12 The normalized intensity of the far-field pattern measured on-axis vs the applied transverse compression stress.

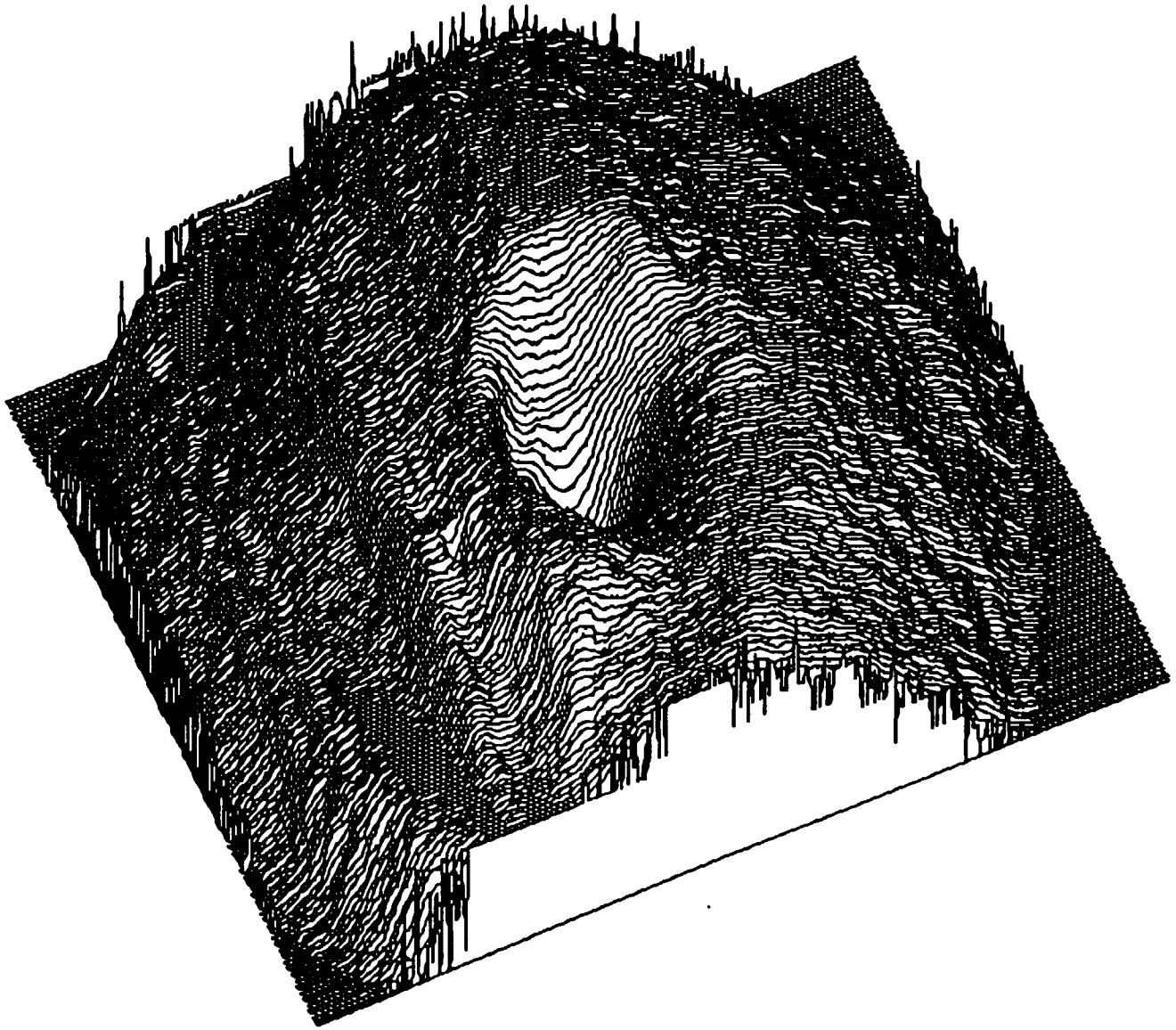


Fig13 A sample of a 3-D surface image of the fiber far-field pattern in the presence of external micro-bending

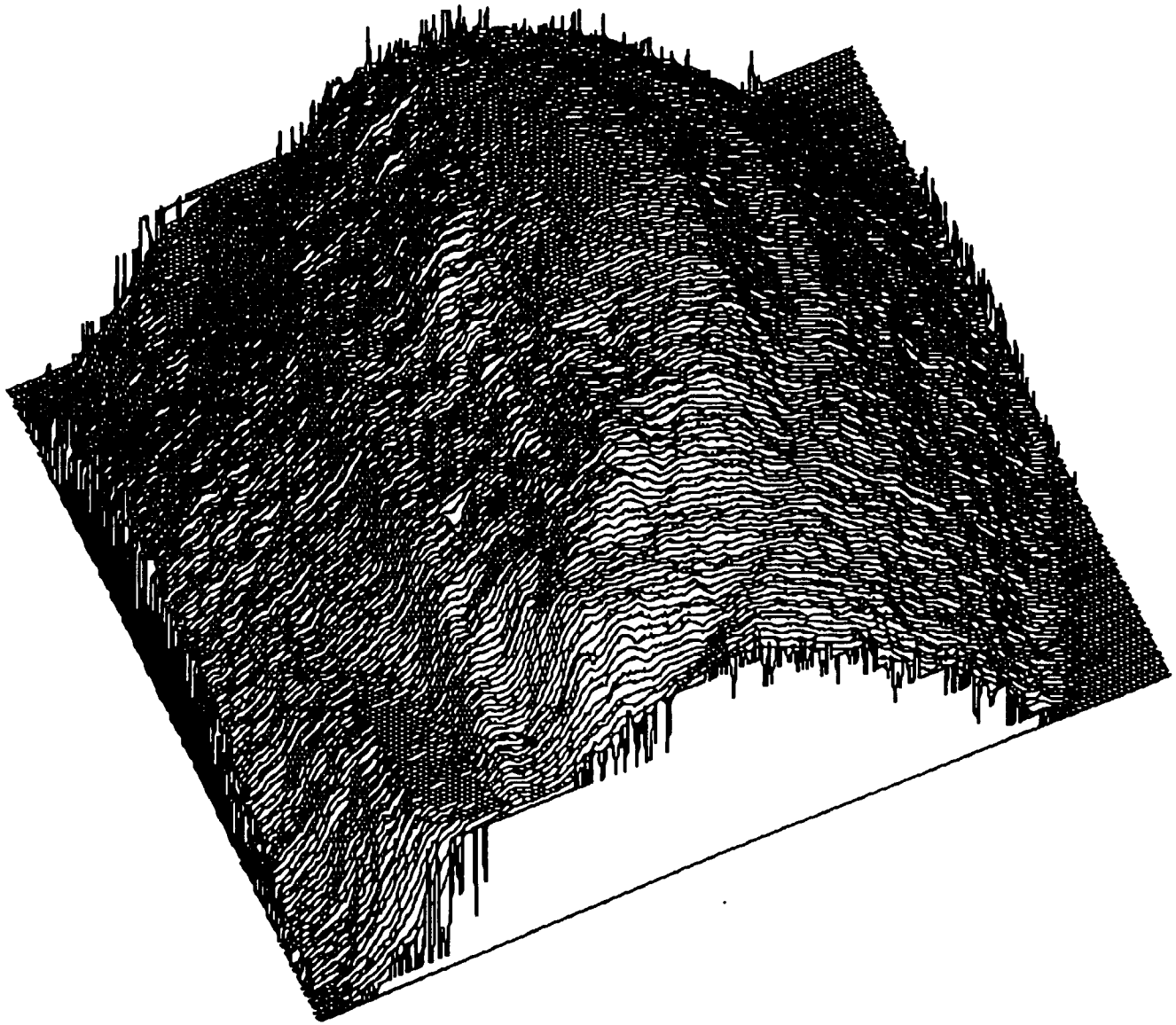


Fig. 4 A sample of a 3-D surface image of the fiber far-field pattern before applying external stress.

The Semiannual Report

of

A Research Grant Award

1. **Number:** **NAG3-1347** **Basic Award**

2. **To:** Drexel University
Philadelphia, PA 19104

3. **For research entitled:**

"Thermal Durability of Sapphire Optical Waveguides Processed Into High Temperature Ceramic Composites."

4. **Under the direction of (Principal Investigator):** Dr. M.A. El-Sherif

5. **Duration:** One (1) Year

6. **Beginning date:** April 1, 1992

7. **NASA Technical Officer:** Dr. Dan Roth

Address:

Structural Integrity Branch
NASA Lewis Research Center
Mail Stop 6-119
21000 Brookpark Road
Cleveland, OH 44135

8. **Date of report:** October 15, 1992

Contents

INTRODUCTION

Conventional optical fibers used for transmission of information and sensor applications are made of silica (typically 125 to 140 mm diameter) and are coated with acrylic polymers. At very high temperatures (over 1000° C), sapphire fibers offer an alternative technology for sensor applications. Sapphire (Al_2O_3) fibers possess reasonable optical propagation properties and can potentially perform without significant degradation up to 1500° C. For this project, a protectively-coated sapphire fiber is proposed for intrinsic sensors embedded in ceramic composites. The work plan is focused on:

i. Design and fabrication of protectively-coated optical sapphire fibers;

ii. Processing of ceramic matrix composites containing optical fibers, and

iii. Fabrication and testing of samples before and after thermal cycling.

A comprehensive study was performed on sapphire fiber waveguides (geometry, properties, and cladding and protective coating materials). The study also included a detailed investigation of different techniques for coating the waveguides, as well as fabrication. The quality of the interface contacts and adhesion between successive layers that can be achieved during the coating process are very important. Therefore, a surface treatment is required in order to promote a strong bond between the layers at the interfaces. A developed chemical deposition technique was applied and experimentally tested. An optical grade single crystal sapphire fiber is used as the core of a multimode optical fiber. This sapphire fiber is coated with a

thin layer of polycrystalline alumina by introducing very fine alumina particles (20 nm) in a polymerizable monomer carrier. The monomer is allowed to polymerize, leaving a uniform coating on the fiber surface. The polymer, polyacrylic acid, is compatible with the alumina surface, giving good adhesion to both the particles and the fiber surface. Binder removal and subsequent sintering were performed by heating the coated fibers to 600°C and ~1300°C, respectively. This last process was optimized in order to eliminate porosity and insure coating uniformity. This thin layer of fine particles provides the necessary bonds between the optical fiber core and the cladding material. For the cladding material's deposition, and to reduce the number of interfaces in the structure of the multilayer fiber, the same deposition technique is repeated to grown the cladding layer directly on the fiber core. In this way, the necessary requirements, for strong bonds with the fiber core, is achieved. Several samples of single crystal sapphire fibers were coated and examined under a SEM.

The second task intails processing of ceramic matrix composites containing the optical sapphire fibers. Several processing techniques were examined. As a result of this study, the experimental investigation was focused on two promising techniques, as explained below:

a. Currently, there is no quick and easy way to produce a ceramic green body having an optical fiber embedded with exposed leads, as protecting the leads of the optical fiber during the impregnation process is a major concern. In preliminary tests, a few samples were produced using conventional manual methods, however, they had flaws and cracks upon drying due to the lower density of the green bodies. If the specimen is compressed in a mold specially designed to remove the excess water, a

dense body could be made. This mold would allow the optical fibers to be embedded during the forming process, and because of the even pressure exerted during compression, major flaws in the material would be avoided. Therefore, the mold will include a stainless steel filter to release excess water. Details of the structure will be discussed later.

b. A colloidal approach (slip casting) is used to process the samples. Alumina slurries are prepared and poured into a specially designed gypsum mold for consolidation. The optical fibers are incorporated into the slurries by mixing or squeezing with the ends of the fibers exposed. The consolidated samples are then heat treated at around 1400° C for several hours, densifying them.

During the course of this work, several difficulties arose in determining key parameters required for the coating and impregnation processes. Promising techniques are now in progress, as will be discussed in detail later in this report. As a result of these difficulties, the time needed to complete these tasks will exceed the proposed time. Consequently, the work on task three (Testing: series A,B) will be shifted a few weeks and completed during the fourth quarter of the program, as shown in Table 1. The amount of optical grade sapphire fiber used for experimental investigation was much higher than estimated. This has resulted in shortages of sapphire fiber for the rest of this program. These fibers are special high quality optical fiber provided by MIT. To overcome this problem, and, at the same time, satisfy the technical goals proposed for this project, we have requested that the number of optical fibers in each of the required specimens be reduced from five to two pieces, one oriented on the axis of the specimen and the second oriented on the side of the specimen, as presented in the proposal.

Transferred data from the optical fibers will reflect the properties of the materials near to the surface and deep in the bulk. This point has been discussed technically with Dr. Dan Roth, the NASA Technical Officer, and we appreciate his approval on this minor modification.

Regardless of these difficulties, work on coating the optical fibers has been successfully completed. Several techniques were investigated and a newly developed technique, using polymerized suspension was preferred. The results show a very high quality deposition of an alumina cladding layer on a single crystal sapphire fiber core. The work on this task has been completed successfully and the results have been accepted for publication in the 17th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach January 10-15, 1993. A copy of the acceptance letter and abstract are included in Appendix A. The coating process and the critical parameter will be discussed in report. Also, a brief description of the progress made concerning each of the tasks are presented herein:

OPTICAL FIBER DESIGN AND FABRICATION

Optical grade sapphire fibers are grown as high quality single crystal fibers. In contrast to conventional silica based optical fibers, which have cladding layer of a lower refractive index than the core materials, these alumina fibers are uncladded. Such cladding layers are required to confine the propagating optical signal within the fiber core and limit attenuation and losses. Also, such sapphire fibers do not have external layers designed to protect the fiber from its surrounding environment. These types of protective coatings have

been shown to be of special importance in applications involving high temperature ceramic composites.

Two layers of coating on the optical sapphire fibers are necessary. A cladding layer of alumina or silica and a protective layer of silicon carbide grown on the optical fibers will confine the optical signals and protect the fiber over a large range of temperature, pressures and environmental conditions.

The conclusions drawn from the study performed on the design of protectively-coated sapphire fibers are as follows:

a. For thermal durability of the sapphire optical waveguide, alumina is the preferred cladding material, whereas silica (SiO_2) will provide better confinement of the optical signal within the core. Since the emphasis of this project is on thermal durability, alumina will be used as the cladding layer of the proposed sample. The thickness of the cladding layer will be in the order of 20-30 μm.

b. A second layer of coating using silicon carbide as a protective layer is preferred to other materials due to the mechanical and thermal properties of SiC and the properties of the ceramic matrix Al_2O_3. A thin layer of SiC will be grown on top of the cladding layer. This coating layer will prevent long term deterioration of strength and stiffness of the optical fibers at elevated temperatures. This layer will also limit the sources of surface damage existing at the matrix/fiber interface.

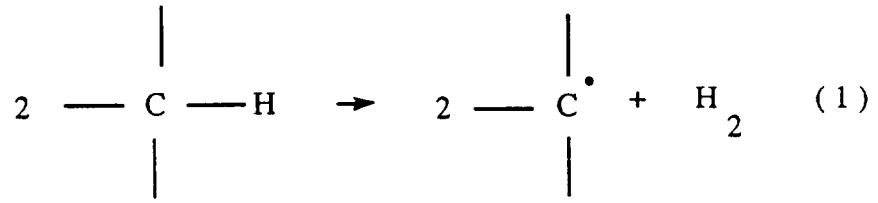
I. Coating and Surface Treatment of Sapphire Fiber

I.1. General

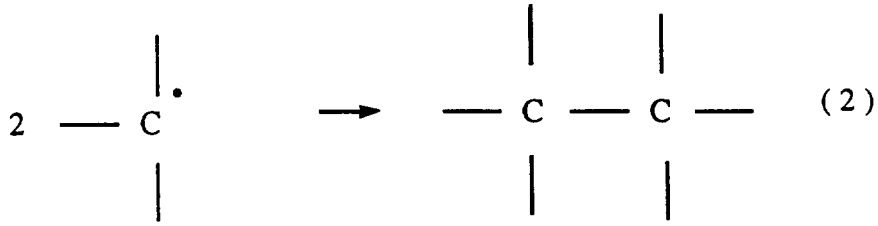
The main objective of this task is to design and fabricate a protectively-coated sapphire fiber. The types of the coating material is important for the transfer of strain and temperature from the ceramic matrix materials to be evaluated to the sapphire fiber sensor element. In high temperature measurement applications, a coefficient of thermal expansion mismatch between the sapphire fiber element and the coating layers may lead to a loss of interfacial contact and result in discontinuity in heat flow and strain distribution at the boundaries. Thus, the interface contact quality and layer adhesion achieved during the fabrication process, are equally important. For this reason, a novel technique has been developed to grow a layer of very fine aluminum oxide particles (nanosize particles having a diameter of about 20 nanometer) on the surface of the fiber, using a chemical deposition technique. This layer of fine alumina particles will act as the cladding layer. After completion of the coating process the fiber was treated to sinter the cladding material.

As a result of work performed in the past six months, a chemical deposition technique compatible with sapphire fibers has been developed and tested. Coating of the fiber with the cladding layer was achieved by placing the fiber for a half hour in a suspension containing PAA (polyacrylic acid) and alumina powder. A polymerized coating containing the alumina powder was applied and then heated to remove the polymer. Further heating at a higher temperature is required for sintering the alumina, forming the cladding layer.

Many common polymers such as polyethylene and polyester are formed by addition and condensation polymerization. Monomers are linked to form polymers by various kind of initiations followed by propagation and, finally, termination. In polymerization, initiation of a polymer can be characterized as atomic or elemental. In this class of polymerization, chemical activators are used to promote polymerization at room temperature. Currently, most chemically polymerizations utilize an amino-peroxide initiating system (for example using the accelerator N, N-dimethyl-p- toluidine [DMPT] and the initiator benzoyl peroxide [BPO]). DMPT is added to the monomer (acrylic acid) and is interacts with the BPO. The product is added to the filler (alumina powder), to produce free radicals on the polymer side chains. These radicals by saturating one another, produce C-C crosslinks. Thus, the cross linking in its simplest terms can be express as follows:



(1)



(2)

However, the cross linking mechanism can sometimes be more complex. In various suspension systems, 0.05% to 2.0% of the initiator DMPT and between 0.5% to 2.0% of the accelerator BPO are used for the monomer polymerization, based on the weight of the monomer. Varying the quantities of the initiator and the accelerator permits adjusting of the working time of the suspension before hardening. This developed technique for coating sapphire fiber will be discussed next.

I.2. Suspension preparation

I.2.1. Materials

i. Tertiary amine accelerator N-Ndimethyl-para-toullidine (DMPT).
ii. Benzoyl preoxide (BPO) initiator.
iii. Acrylic acid monomer.
iv. Aluminum oxide particles ($AL_2\ O_3$) size 20 nanometer.

I.2.2. Procedure

i. Study of the monomer properties

a. 0.1 to 0.5 wt% DMPT (based on the weight of the monomer) was used to determine the best concentration of the initiator in the mixture.

b. A measured amount of BPO was dissolved in acetone and added to the mixture at a concentration of 0.1 to 0.5 wt % BPO, based on the weight of the diluted monomer only. The powder was allowed to dry at room temperature to remove the acetone.

c. Up to 0.05 gm DMPT and 0.05 gm BPO was added to 10 ml acrylic acid and 10 ml H_2O. The hardening time, controlled by the levels of initiator and accelerator was studied for each solution concentration.

ii. Preparation of homogeneous suspension

As a result of the study performed in part (i) and according to the monomer properties required, it was decided to prepare a suspension of 0.05 gm DMPT and 0.05 gm BPO added to 10 ml acrylic acid and 10 ml H_2O. The hardening time required for this mixture is between one hour and half to two hours. Aluminum oxide (Al_2O_3) particles were added to the solution at a loading level of 1gm (10 wt %), 1.5 gm (15 wt %), and 2 gm (20 wt %) and the powder was mixed well to have a homogeneous solution.

II. Fiber Coating

a. An optical grade single crystal C-axis sapphire fiber prepared at MIT using a laser heated floating zone process was used in the application. The fiber was cleaned by immersing it in HCl for half hour and washing it in distilled water. The fiber was then immersed in the prepared suspension for half hour for deposition of a coating layer on the fiber surface. The fiber is then taken out of the suspension and left for 24 hours to polymerize in room temperature.

b. Changes in fiber surface morphology as a result of surface treatment and coating were studied by SEM.

c. The coated fibers were heated to 600°C for varying times (10 to 60 minutes) to remove the polymer from the cladding layer. The temperature was then increased to 1300 up to 1500° C (10-60 minutes) to sinter the alumina particles. The heating and cooling rates for the furnace were in 2-10° C/min range.

d. At each stage of the process, the coated fibers were tested and studied using a scanning electron microscope; the results are presented next.

III. Results and Discussion

The original single crystal sapphire fiber was examined by the SEM. The results shown in Fig. 1 indicate a very smooth surface for these types of fibers. After the fiber was immersed in 10% aluminum oxide suspension for a half hour, the fiber was coated by a layer of polymer and the alumina powder as shown in Fig 2. The fiber was heated to remove the polymer and sinter the alumina powder around the fiber. For the first trails a very thin coating layer was achieved; however, this layer was not homogenous (fig 3). As a result, a suspension containing 15% alumina particles was used. Figs 4 and 5 indicate fiber surface morphology before and after heating. The 15% solution still had the same shortcomings. Finally, the same process was repeated with a suspension having 20% alumina powder. The coated fiber surface is shown in (fig 6). The fiber was heated to 600° C for 10 minutes for binder removal and to 1300° C for 10 minutes to sinter the alumina (fig 7). It was clear that the materials were not homogenous and the sintering process was not completed. Therefore, the coating process was repeated and the fiber heated to 1400°C for one hour to sinter the alumina particles. In order to determine the critical temperature for this process, the same process was repeated at higher temperature. The final heating temperature was increased to

1500°C for one hour. The results obtained for the samples heated at 1400°C and 1500°C are show in (fig 8 & 9) respectively. Fig (8) shows clearly that the alumina particles were combined. However, the results shown in fig (9) indicate that the alumina particles have started diffusion at 1500°C . At this point, it is important to examine the behavior of the optical fiber at different temperatures. One single crystal sapphire fiber was heated to 1400° C; another, to 1500° C. The surface morphology for these fibers are shown in figures 10, 11, 12 and 13. These figures show that the surface morphology of the fiber has started to change at 1500°C. Therefore, as a result of this investigation, the maximum temperature for our experimental investigation will be limited to 1400°C.

During the course of this task, several difficulties arose in determining key parameters required to optimize this process, such difficulties are:

a. How can a homogenous suspension containing alumina powder be prepared ?

b. What are optimal concentrations of initiator and accelerator in suspension to change conditions for polymerization?

c. What is the time needed to grow a uniform layer of the polymer/alumina mixture on the surface of the optical fiber?

d. What are the time and temperatures required for heating the fibers to remove the polymer completely and to sinter the alumina particles in the cladding layer?

All of these questions and others were addressed during our investigation to optimize the coating process.

Finally, in order to increase the cladding layer thickness to about 20 mm, the fibers were coated with three layers of the prepared suspension. These fibers

were heated to 600°C for one hour to ensure that the polymer was completely removed. The temperature was increased to 1400° C for an hour for perfectly sintering the alumina powder on the surface of these fibers. An example of each of these cases is shown in Figs 14 & 15, respectively.

In conclusion, a suitable technique for homogenous coating of sapphire fiber was developed. A suspension containing 20% alumina particles (20 nanometers in diameter) was used for coating the fiber with a mixture of alumina and polymer. The fiber was heated at 600° C for an hour to remove the polymer and the temperature was increased up to 1400° C, sintering the alumina powder around the fiber. In this way, a high quality cladding layer was successfuly obtained.

PROCESSING OF CERAMIC SAMPLES

Our objective is to make a 4 in x 1 in x 0.2 in aluminum oxide body with fibers embedded inside and the ends of fibers exposed. Several approaches for processing of the smart ceramic samples were tried. Several difficulties arose in the impregnation process, however, promising techniques are now in progress. A brief description of these approaches is presented herein:

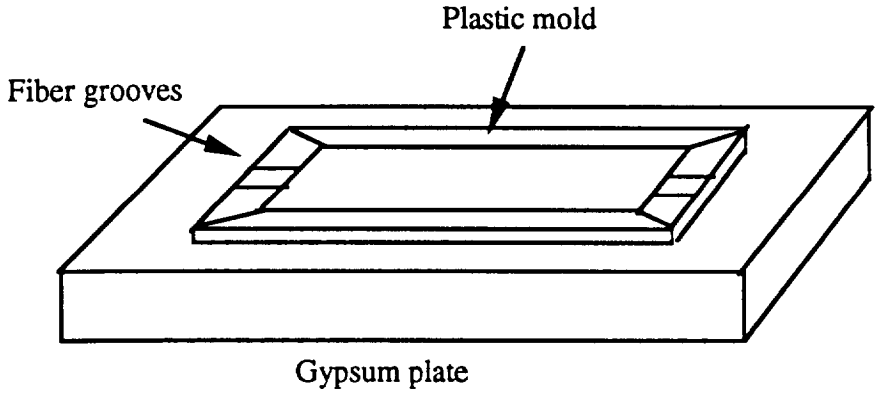
I. Green Body Preparation

Mold

Plaster of Paris was mixed with water in the ratio of 60 to 40. The plaster slurry was poured into a mold made up of cardboard. Before the plaster slurries dried, a 4 in x 1 in x 0.2 in hole was cut out. The mold was then put in a furnace for drying at around 100°C.

Al_2O_3 suspensions

50 vol% aqueous Al_2O_3 suspensions were prepared by first adjusting the pH of the suspensions to 3, followed by sonication using ultrasonics to break up agglomerated powders. After sonication, the pH of the suspensions were adjusted to the desired values. Both pH values of 3 and 9 had been experimented to see whether the dispersed state (pH=3) or flocclated state (pH=9) gives better green bodies. It was found that flocculated suspensions are better in that they are viscous enough to maintain shape after molding. Therefore, the aluminum oxide suspensions were prepared at 50 vol % under flocculated conditions (pH=9). The slurries were poured into the plastic mold sitting on top of a gypsum plate. The two plastic pieces on the side have grooves cut for placing fibers. The set up is shown in the following drawing.



The molded samples were dried in air and then put in a dessicator.

Drying

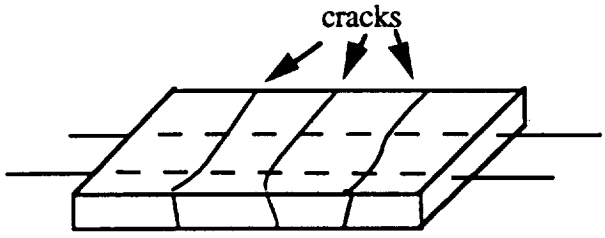
The Al_2O_3 suspensions were poured unto the gypsum mold and were dried in air overnight. It was found that the dried bodies cracked into several pieces. The cracking problem is due to not having enough compaction during the molding process; the samples were too weak to withstand the stresses produced during drying.

Improved approach

Instead of pouring the Al_2O_3 suspensions into the mold, we spread out the suspensions on a gypsum plate. We then compacted and shaped the slurries by hand. The compacted bodies were found to be strong enough to resist drying stresses and no cracking occurred. We also tried squeezing the fibers into the slurries leaving the ends of fibers exposed before the bodies dried. This was also successful and cracking did not occur. The compacted bodies can be cut and smoothed with a knife into the desired size, 4 in x 1 in x 0.2 in.

II. Sintering Study

A molded sample without visible cracks was sintered at 1400°C for 3 hours. Severe cracking occurs after sintering. Cracks formed in the direction perpendicular to the fiber direction as schematically shown below.



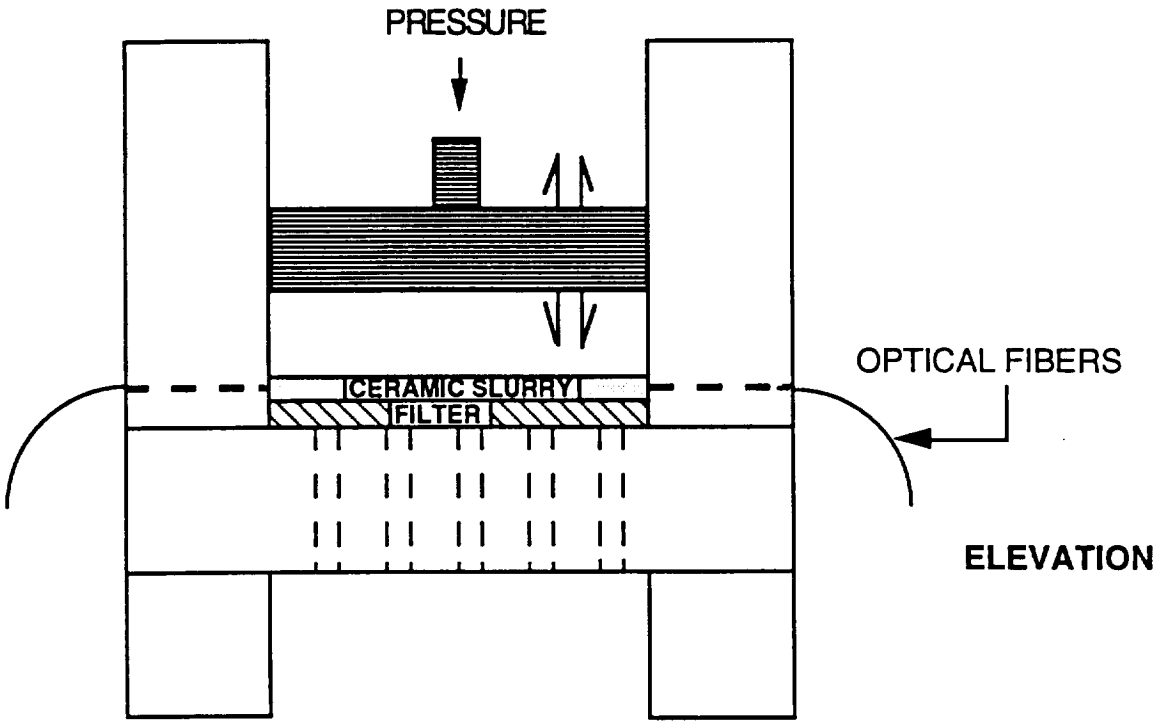
Upon examination of the cross section, it was found that the lower portion was denser than the upper. The higher density of the lower portion resulted from higher green density due to water absorption of the gypsum platet. This gradient in density probably resulted in the cracking of the sintered samples. In addition, the fibers become more brittle compared to fibers prior to sintering. A degree of bending that will break heat-treated fibers will not affect the flexible non heat treated fibers.

III. New Approaches

It was concluded that the main cause for the cracking in sintering is the density gradient and small cracks in a dried green body. To remedy these problems, we took two different approaches.

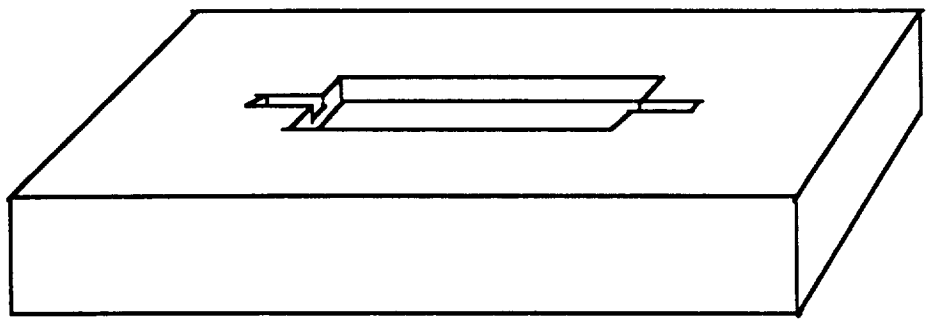
III.1. Pressure filtration

The main advantage of pressure filtration is that uniform high density can be achieved. It is hoped that the cracking problem can be minimized by the pressure filtration. A pressure filtration cell is designed for this application, as shown in the following figure. The pressure-filtration cell consists of a stainless steel filter surrounded by four walls of acrylic plastic. The filter is supported by a plastic plate with holes drilled through it to let out water. Two blocks of plastic raise the cell and a container is placed under the cell to collect filtrate. 4 c-clamps are used to hold the walls of the cell tightly. The pressing of the plunger must be performed slowly to prevent bending of the fibers. During pressing, the fibers are pulled taunt to prevent sagging in the middle of the sample. The shear forces between the wall and the slurry have to be monitored closely in order to prevent the breaking of the fibers.



III.2. Slip casting: gypsum mold with grooves

As mentioned before, the sintered body has higher density at the lower portion of the sample due to water absorption by the gypsum plate. This suggests that if the mold is also made of gypsum with grooves to accomodate fibers, we might achieve high density green bodies. The mold is schematically shown in the following figure.



A top plate made of gypsum will be made to enclose the sample.

NOVEL CONCEPTS

The new processing techniques for coating the cladding layer of the optical sapphire fiber and for the green body preparation and impregnation of ceramic composites containing sapphire fibers, will be considered as a novel concepts. The detailed technical description of these processing techniques will be presented in future reports as soon as the work is completed.

FUTURE PLAN

The dried green bodies will be sintered at temperatures around 1400°C for several hours. The sintered densities will be measured using Achimedes principle as a function of sintering temperatures and time. A suitable sintering program can then be generated. In addition to the pressureless sintering to be studied above, hot pressing will be tested to produce dense Al_2O_3 compacts.

As the work on materials processing is completed, the work on testing and characterization will be started and results will be presented in future reports.

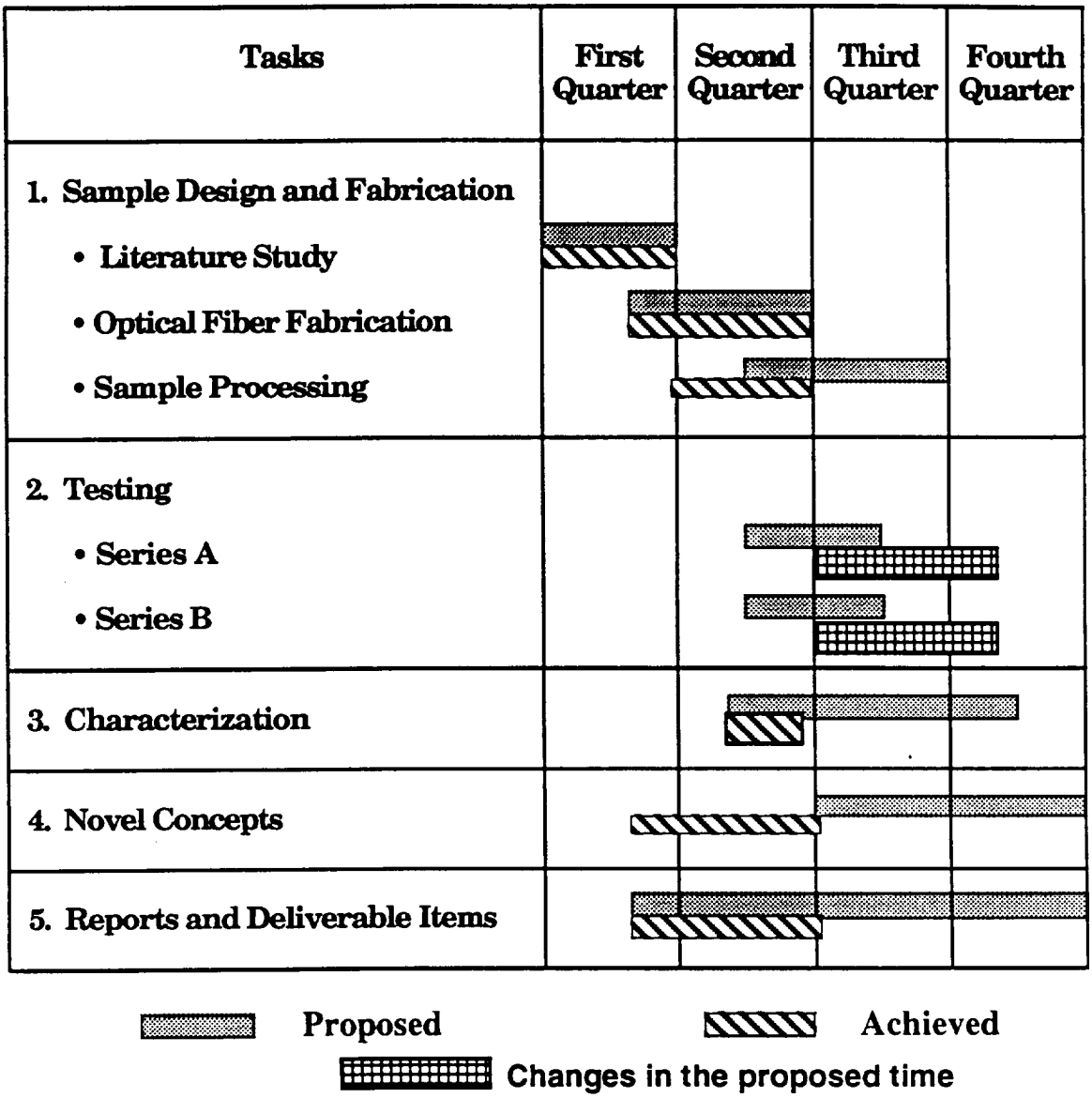
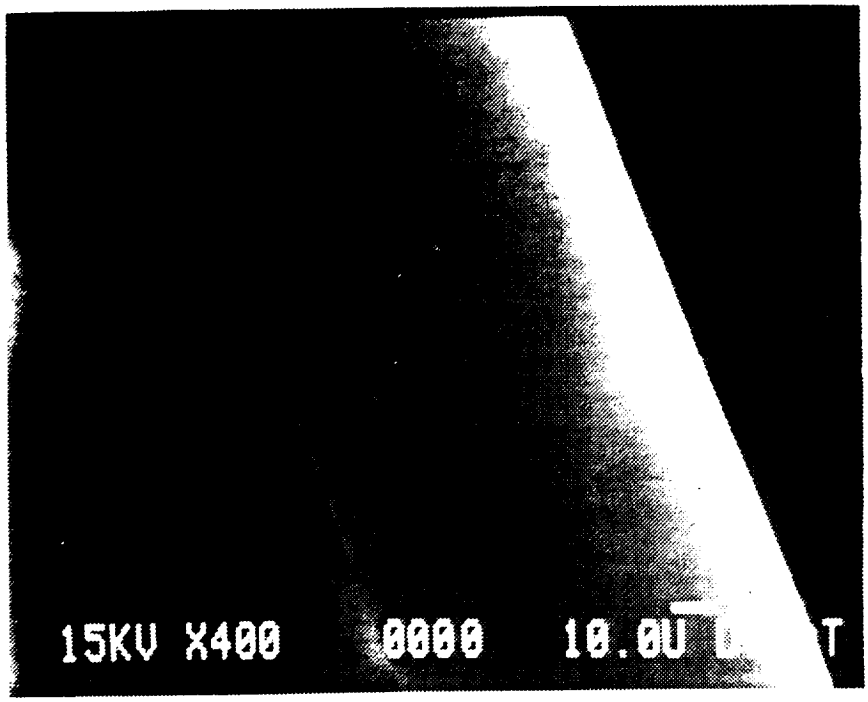
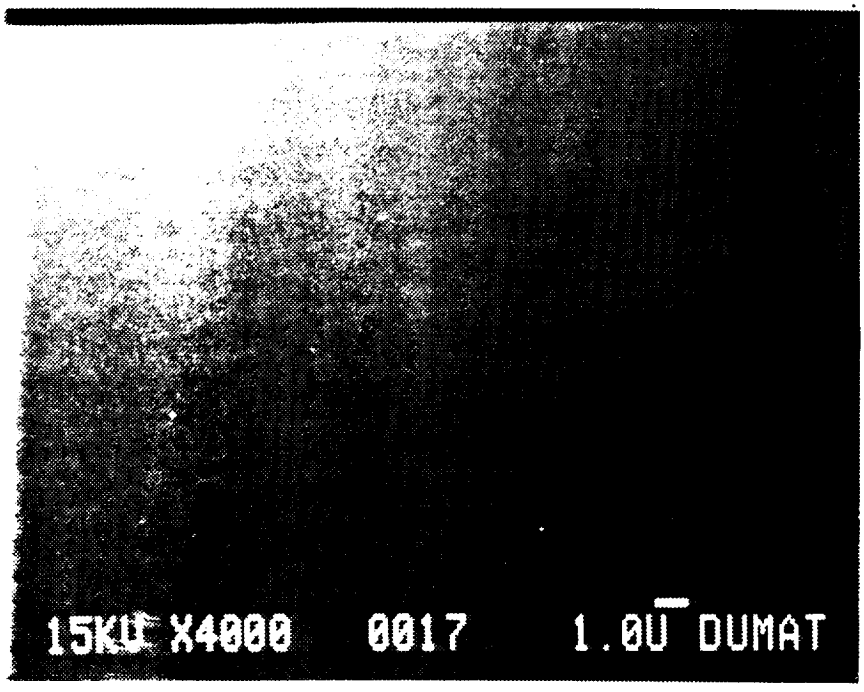


Table 1: The proposed and achieved research work according to the time schedule of the work plan.

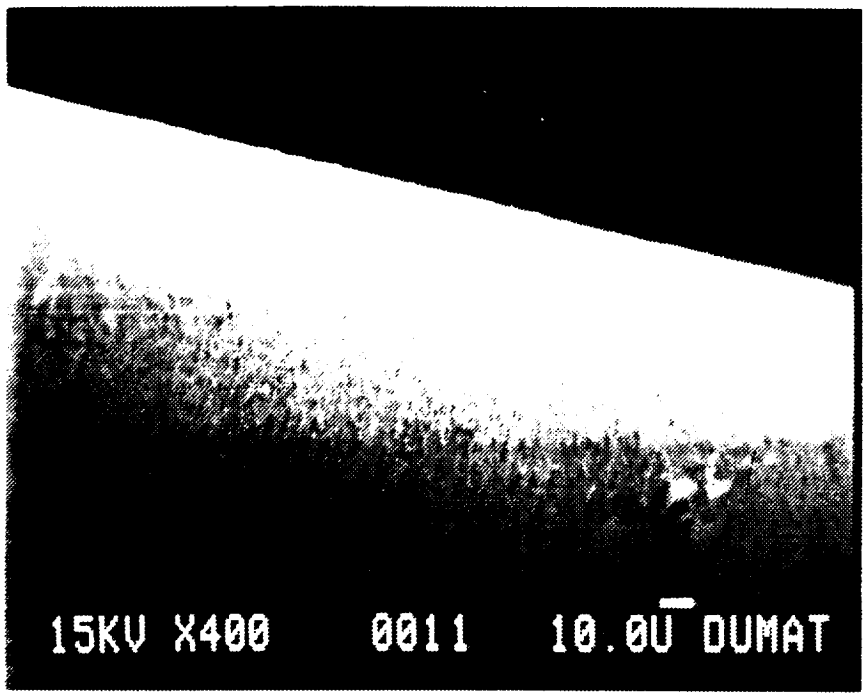


(a)

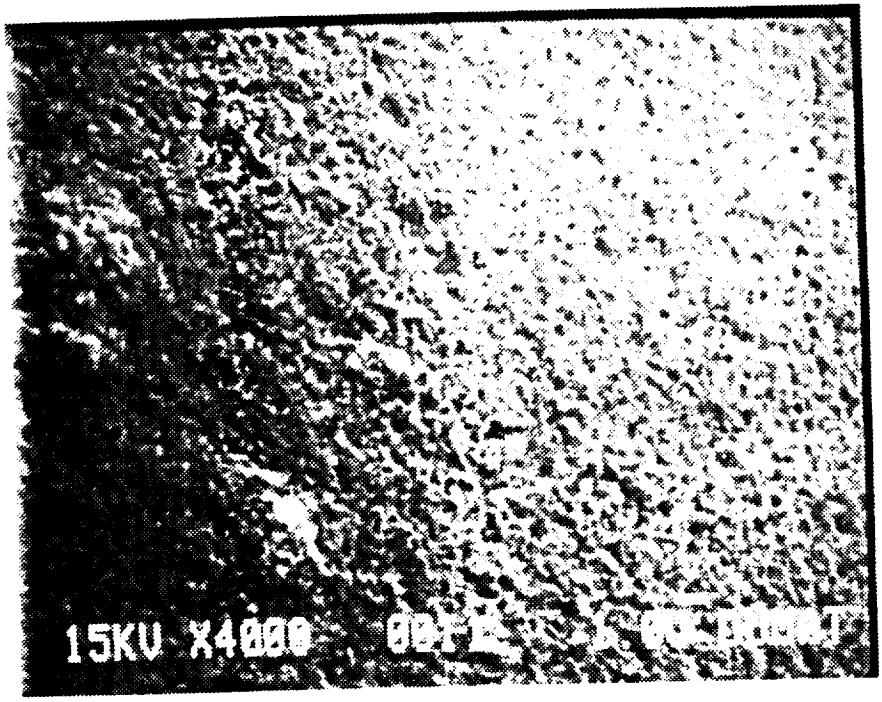


(b)

Fig 1. The original single crystal sapphire fiber (Magnified to (a) 400x and (b) 4000x.)

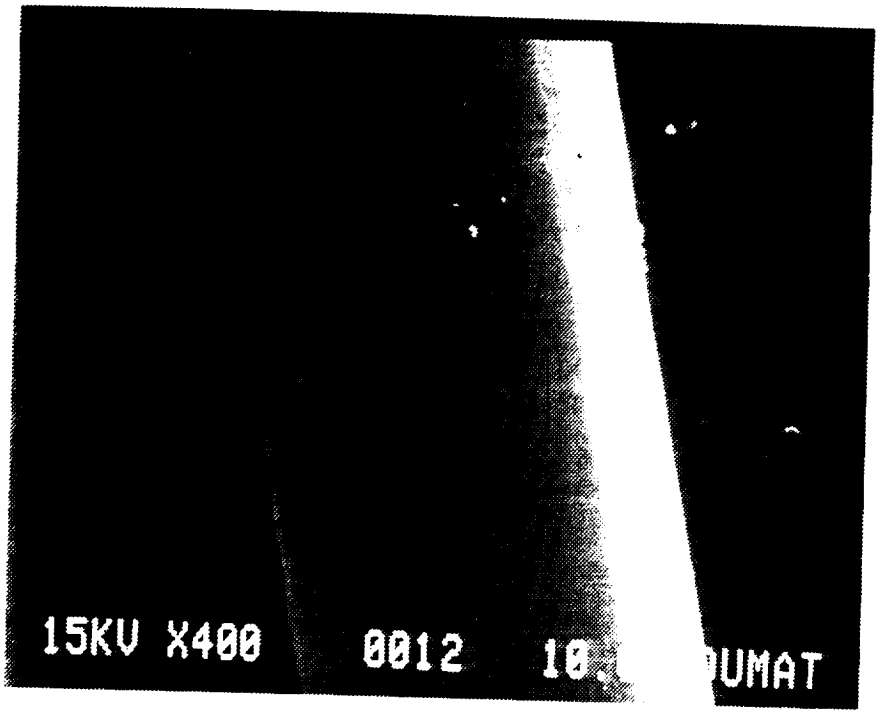


(a)

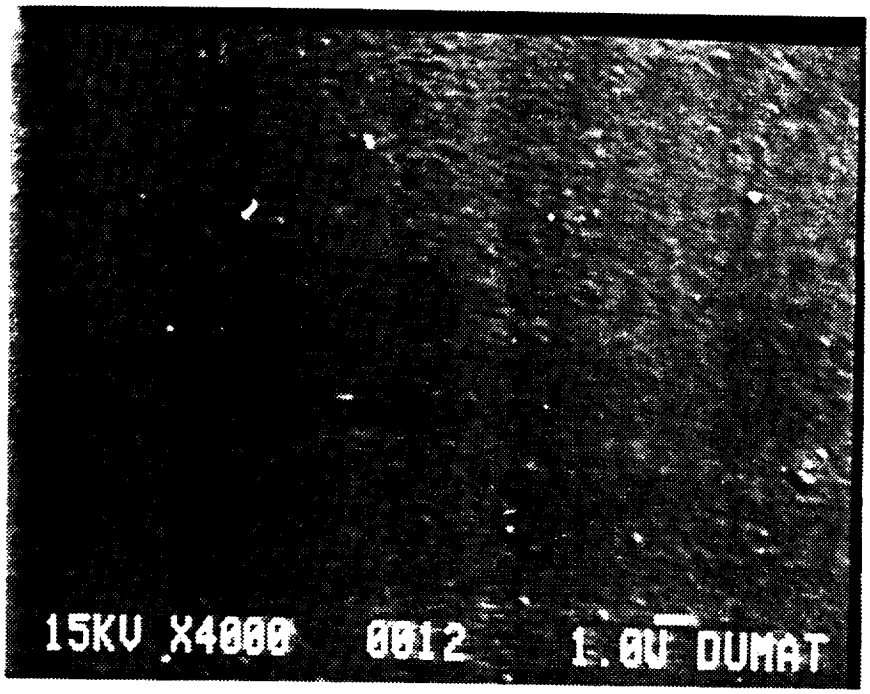


(b)

Fig 2 Treated sapphire fiber in a prepared suspension having a concentration of 10 % alumina at room temperature (Magnified to (a) 400x and (b) 4000x.)

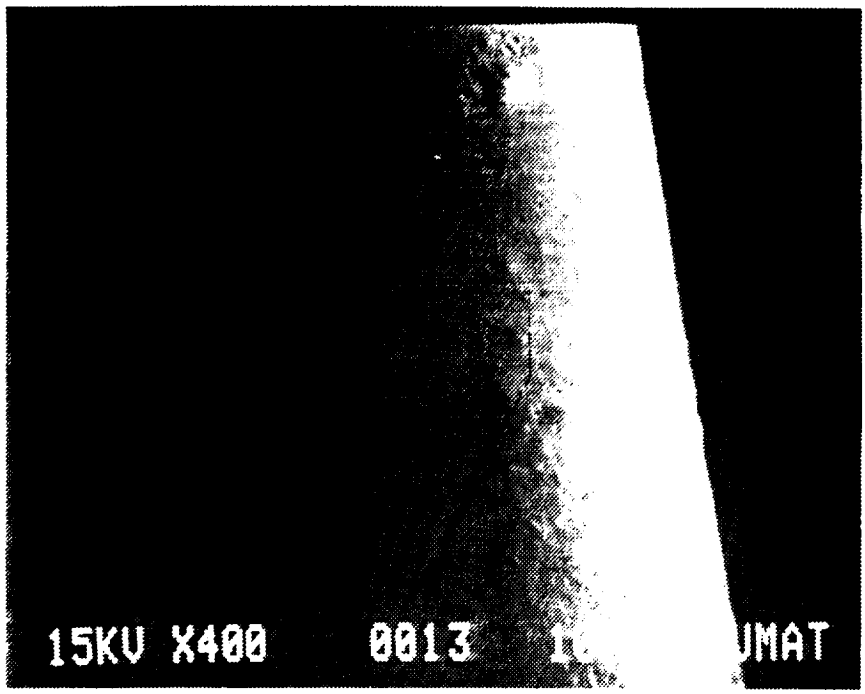


(a)

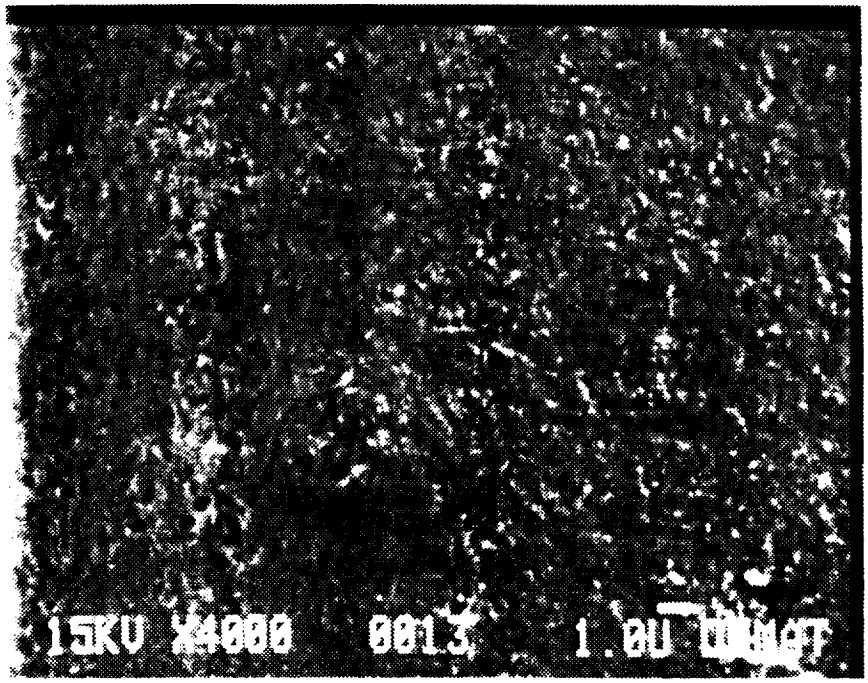


(b)

Fig 3. Treated sapphire fiber in a prepared suspension having a concentration of 10% alumina at room temperature, followed by treatment at 600°C for 10 min. for binder removal and at 1300°C for 10 min. for sintering the alumina (Magnified to (a) 400x and (b) 4000x).

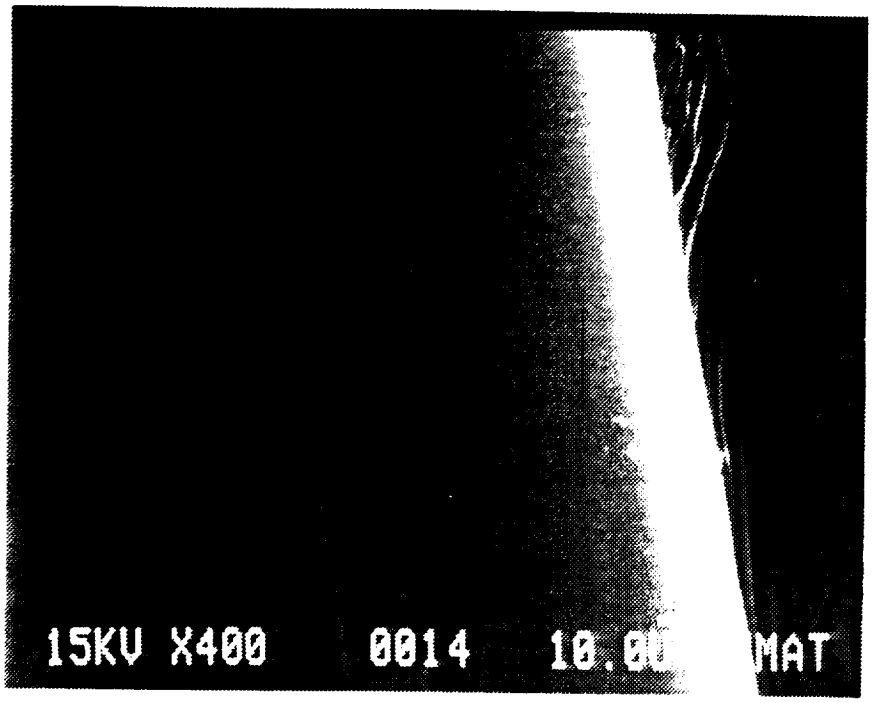


(a)

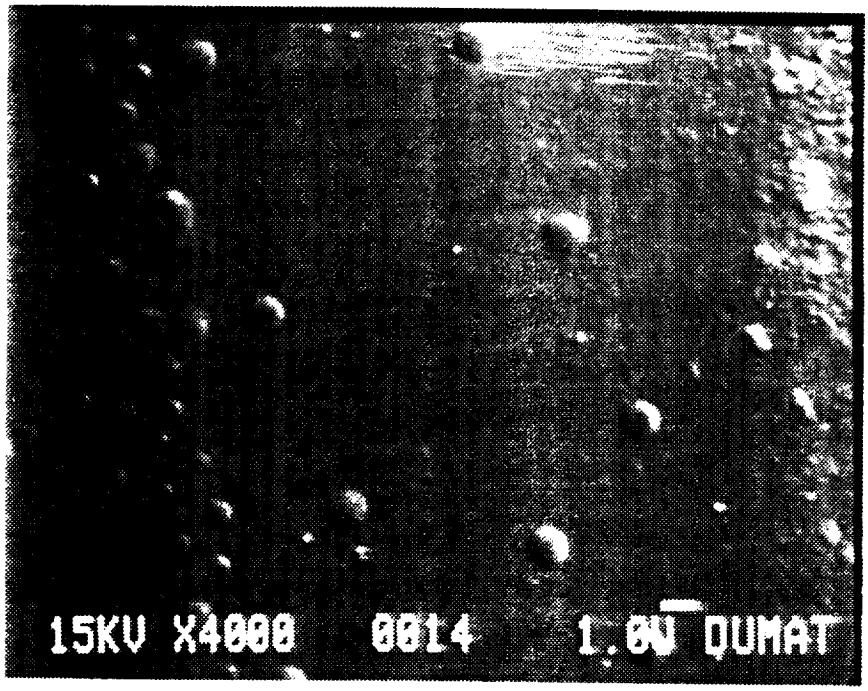


(b)

Fig 4 Treated sapphire fiber in a prepared suspension having a concentration of 15 % alumina at room temperature (Magnified to (a) 400x and (b) 4000x.)

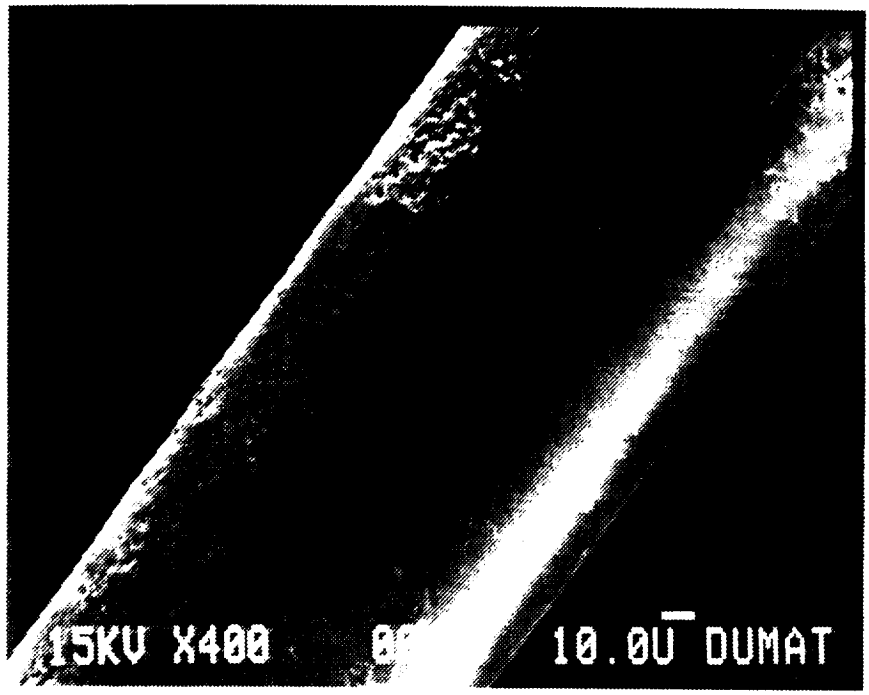


(a)

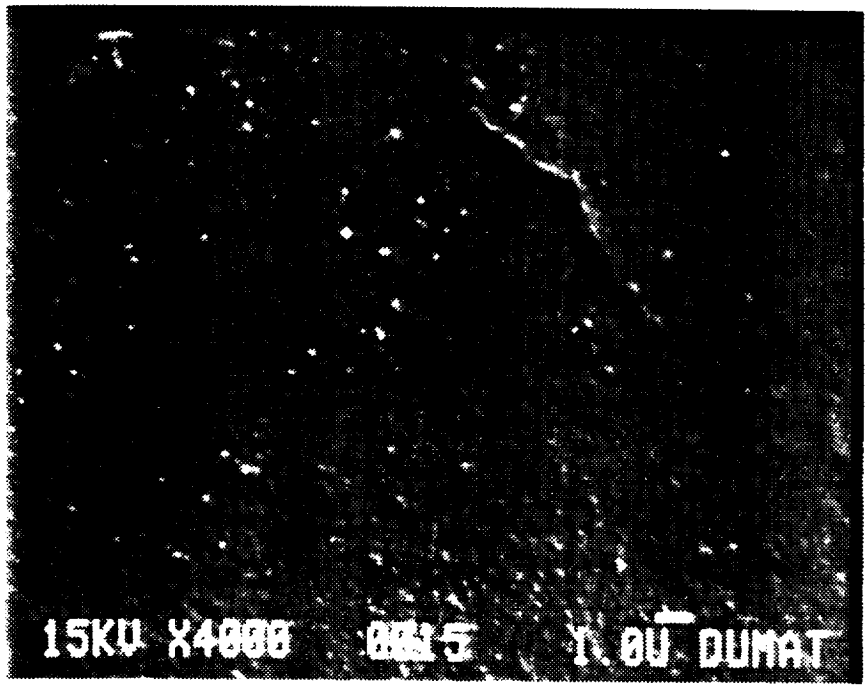


(b)

Fig 5. Treated sapphire fiber in prepared suspension having a concentration of 15% alumina at room temperature. Then thermal treatment was performed at 600°C for 10 min. for binder removal and 1300°C for 10 min. for sintering the alumina (Magnified to (a) 400x and (b) 4000x).

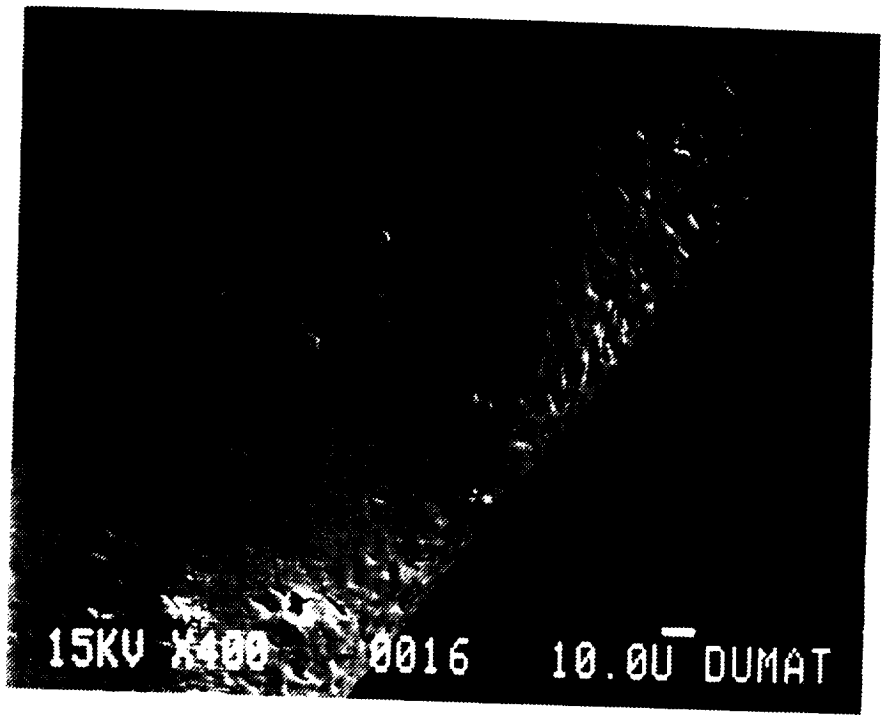


(a)



(b)

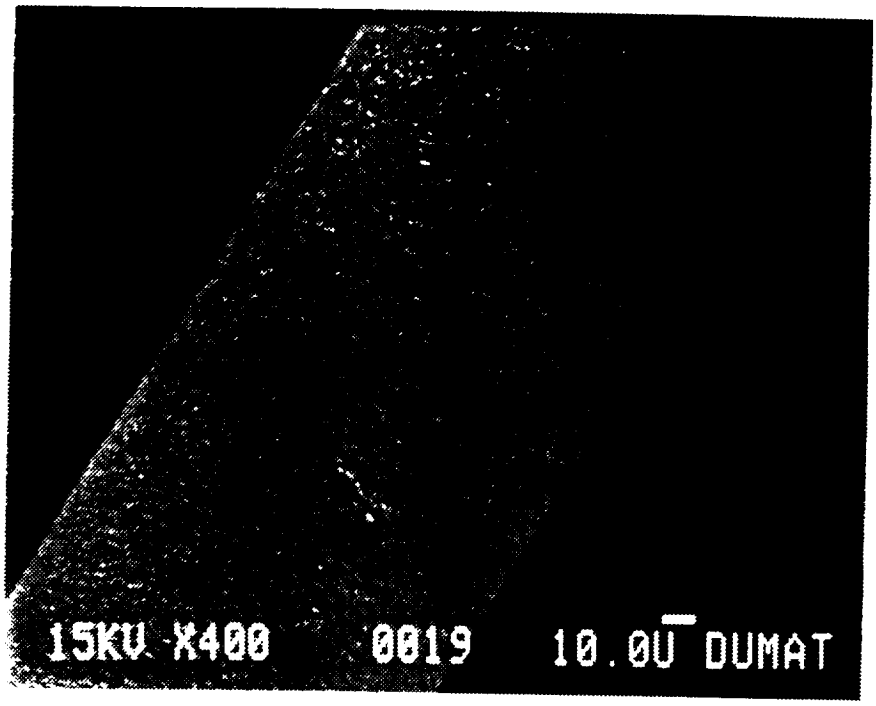
Fig 6. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature (Magnified to (a) 400x and (b) 4000x).



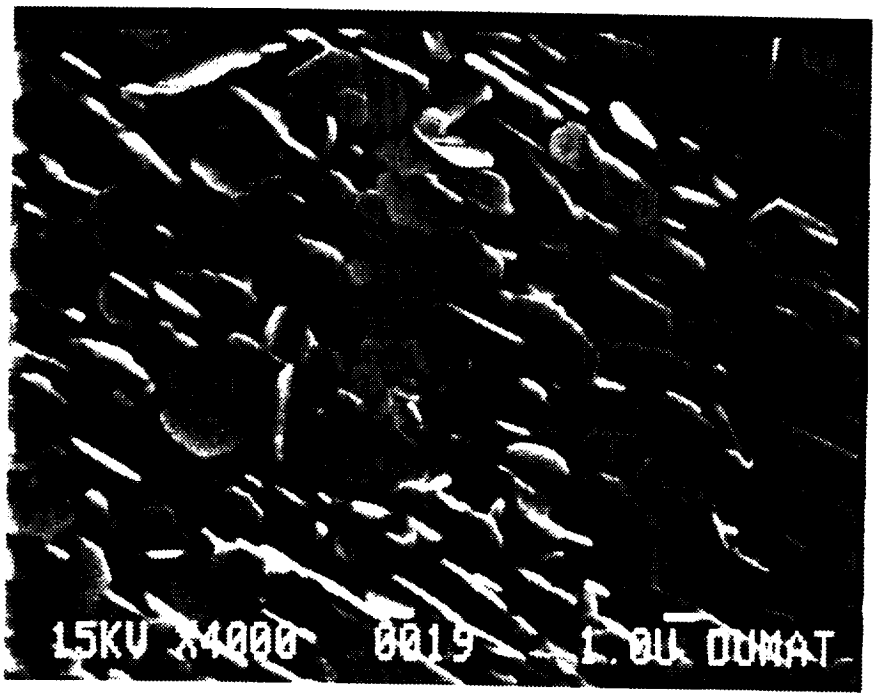
(a)

(b)

Fig 7. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature. Then thermal treatment was performed at 600°C for 10 min. for binder removal and 1300°C for 10 min. for sintering the alumina (Magnified to (a) 400x and (b) 4000x).

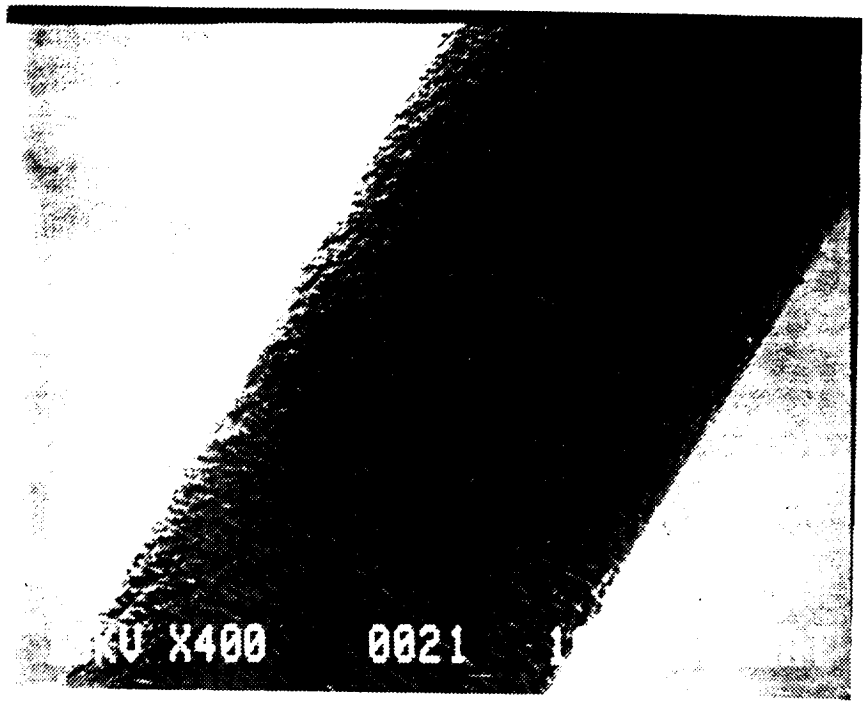


(a)

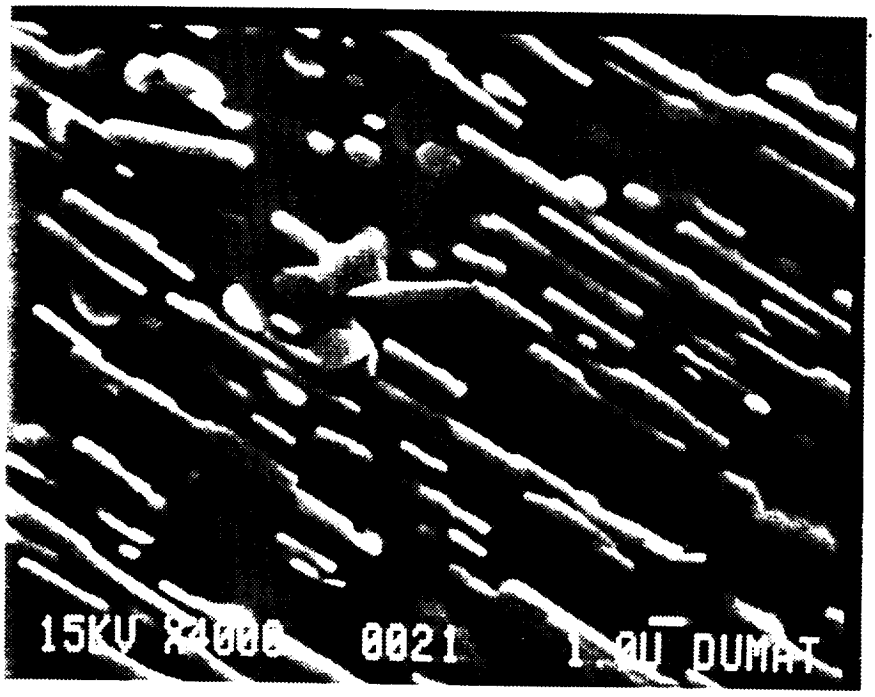


(b)

Fig 8. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature. Then thermal treatment was performed at 600°C for 10 min. for binder removal and 1300°C for 10 min. for sintering the alumina and heated again at 1400°C for one hour (Magnified to (a) 400x and (b) 4000x).



(a)



(b)

Fig 9. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature. Then thermal treatment was performed at 600°C for 10 min. for binder removal and 1300°C for 10 min. for sintering the alumina and heated again at 1500°C for one hour (Magnified to (a) 400x and (b) 4000x).

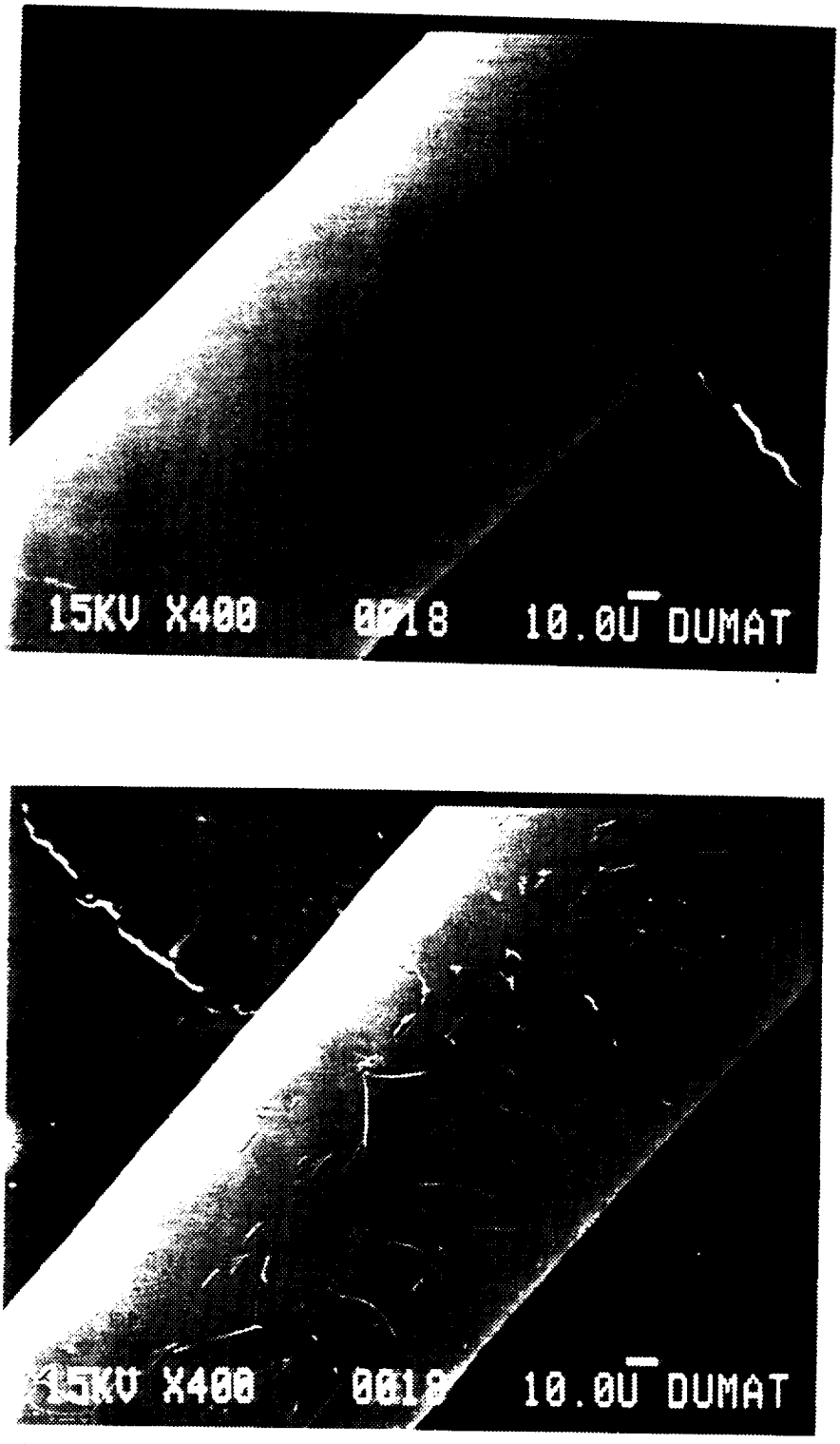


Fig 10. The original fiber heated at 1400°C for 1 hour. The heating and cooling rates were 10°C./min (Magnified to 400x).

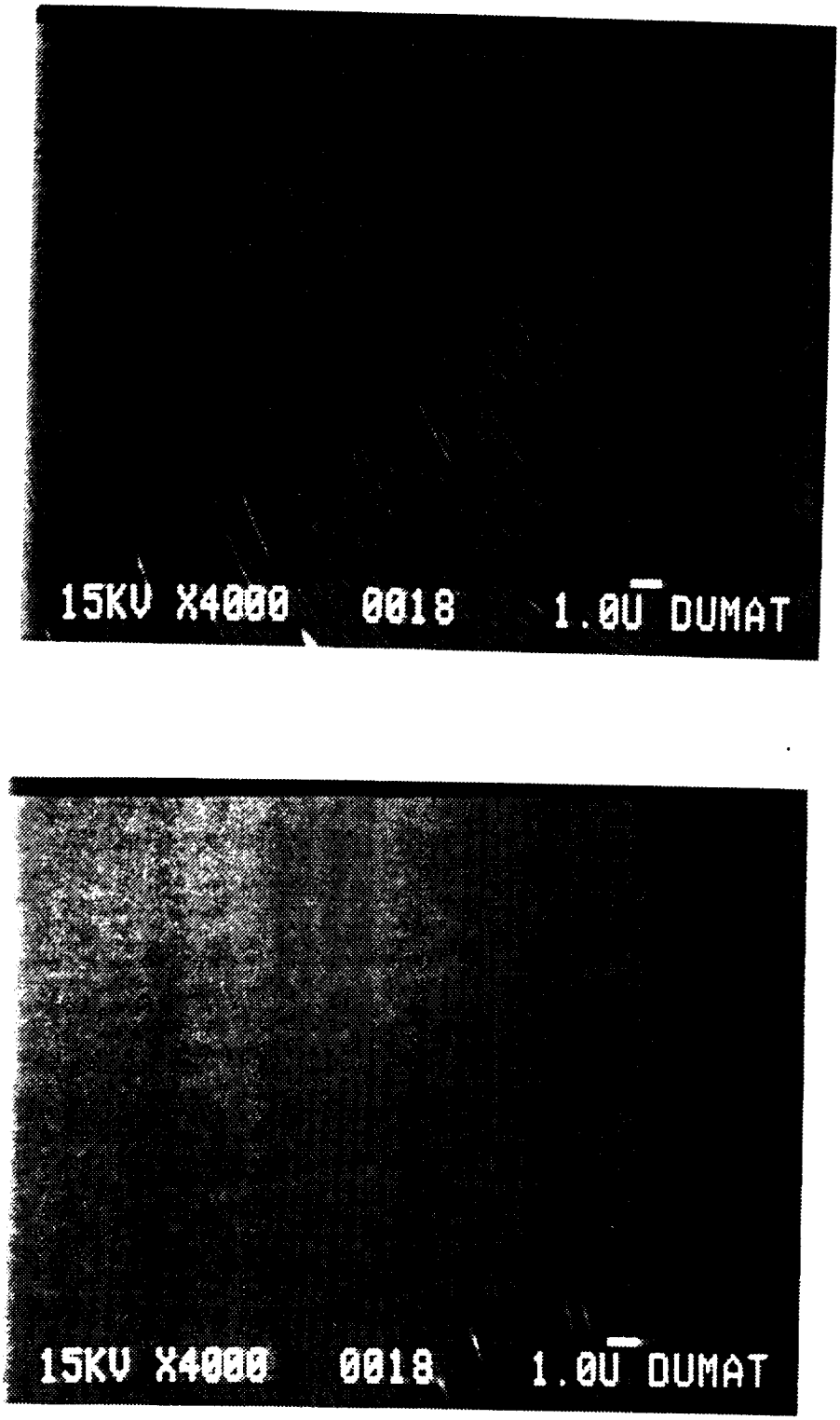
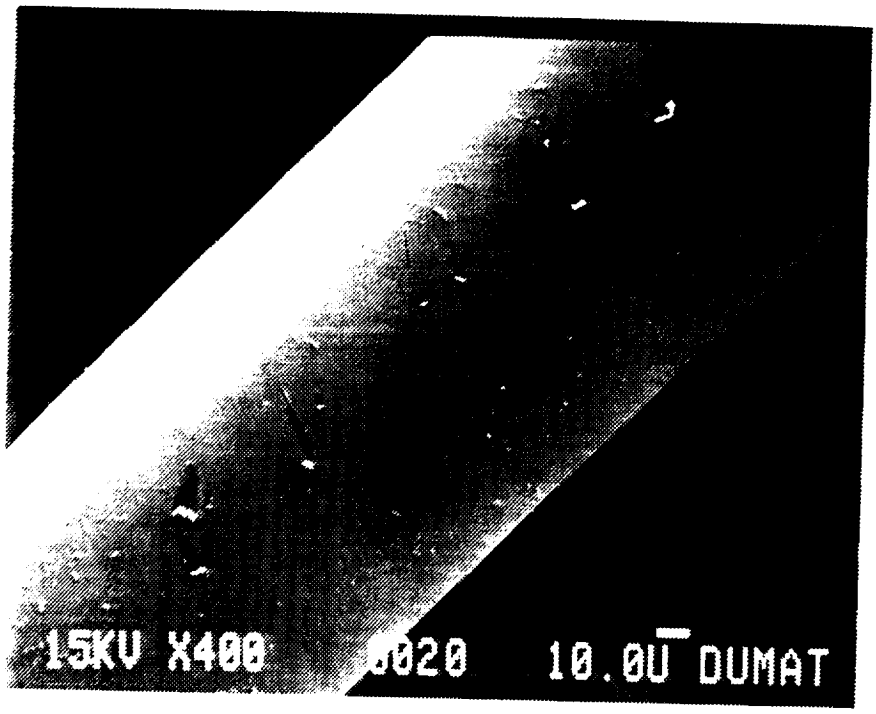


Fig 11. The original fiber heated at 1400°C for 1 hour. The heating and cooling rates were 10°C/min (Magnified to 4000x).



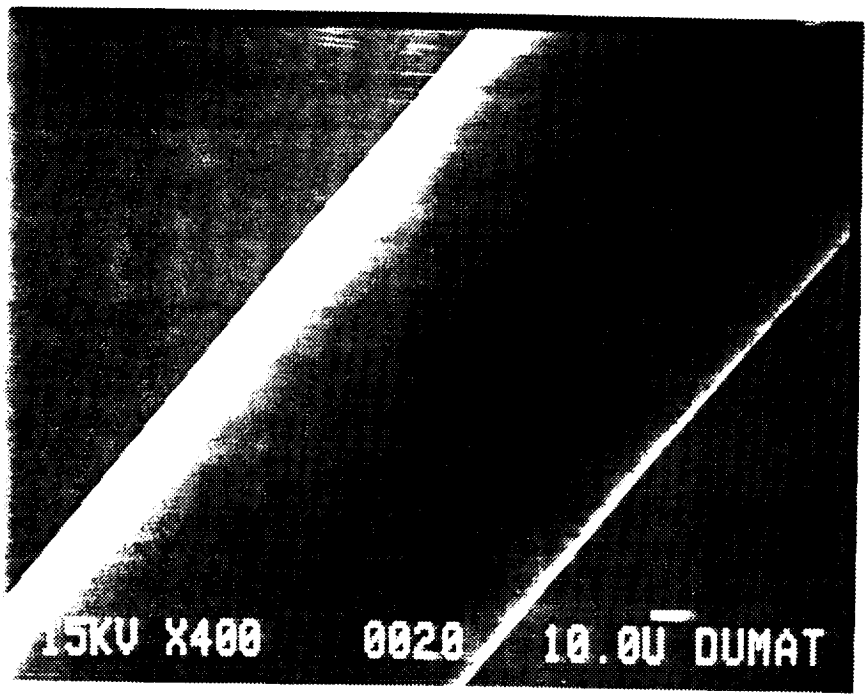


Fig 12. The original fiber heated at 1500°C for 1 hour. The heating and cooling rates were 10°C/min (Magnified to 400x).

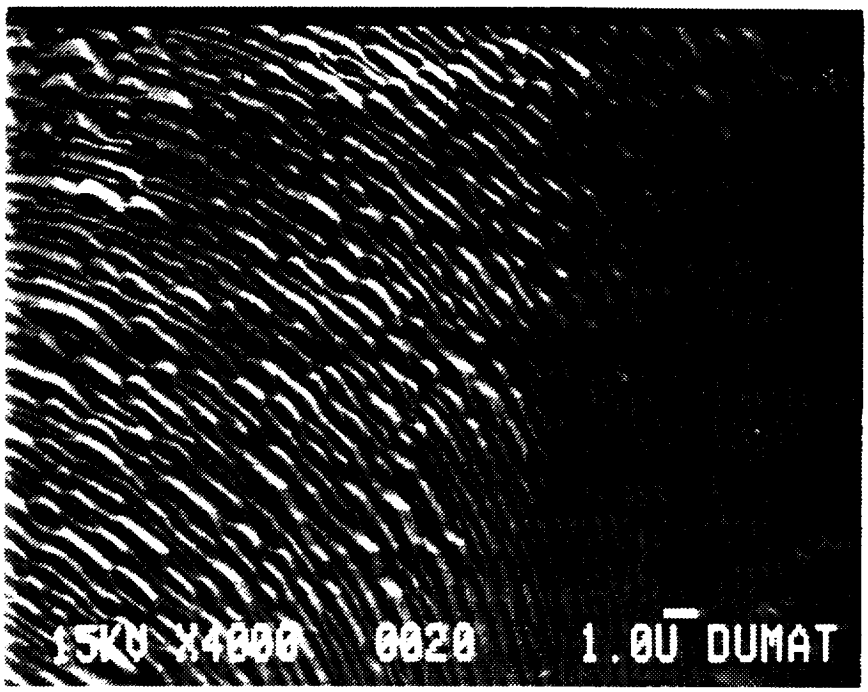
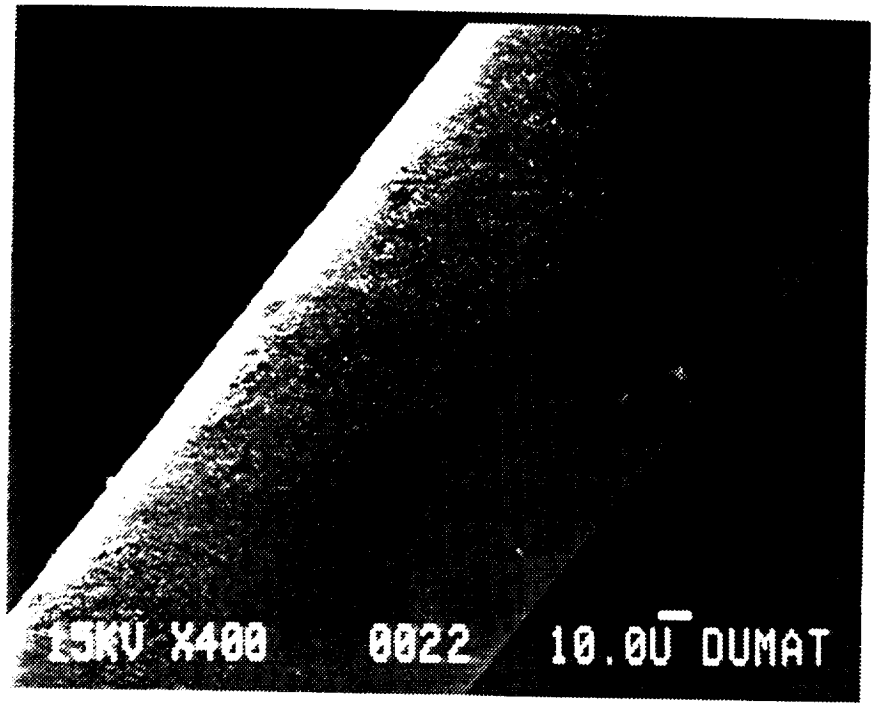
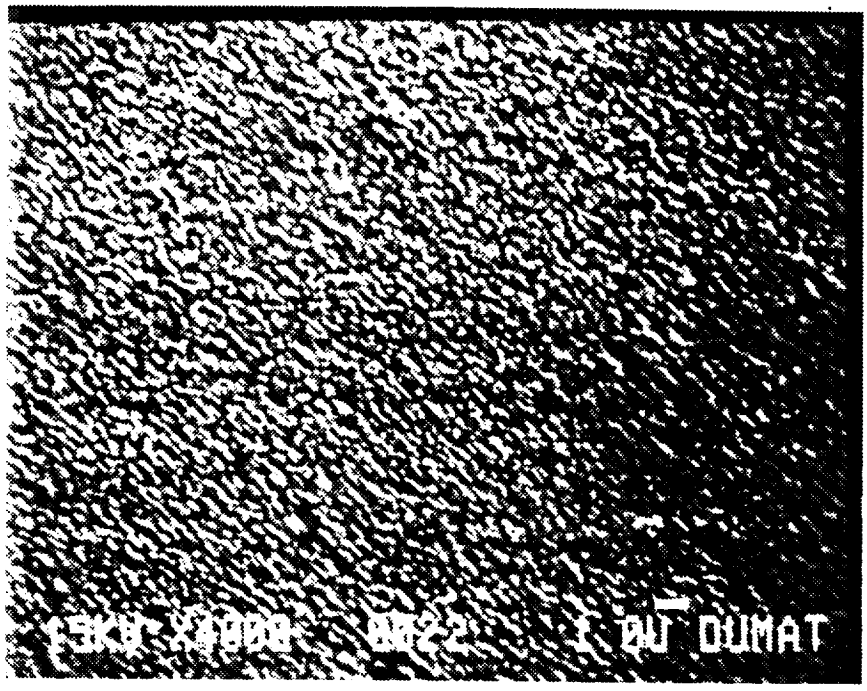


Fig 13. The original fiber heated at 1500°C for 1 hour. The heating and cooling rates were 10°C/min (Magnified to 4000x).

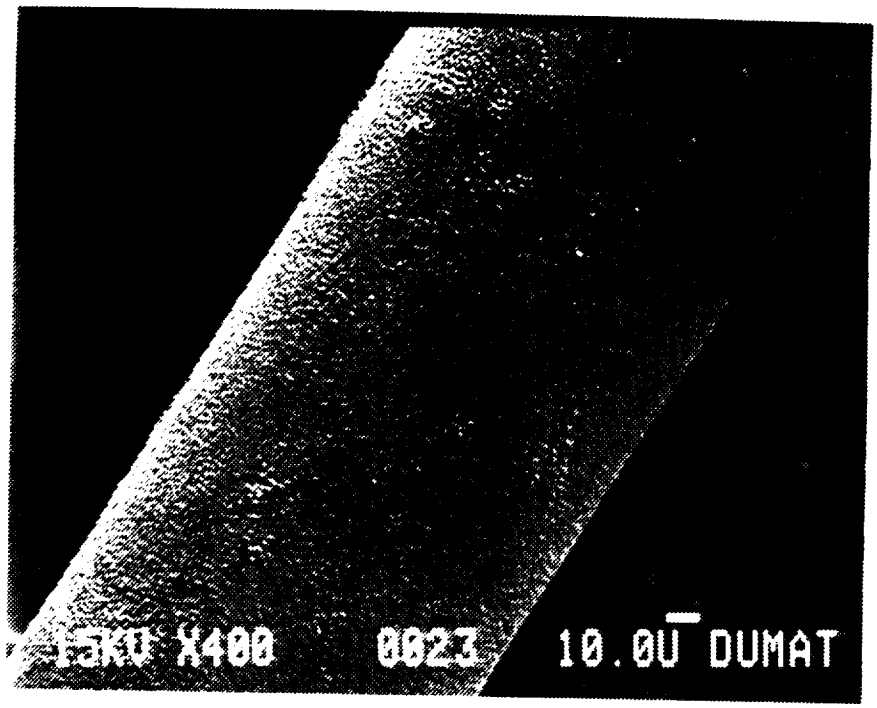


(a)

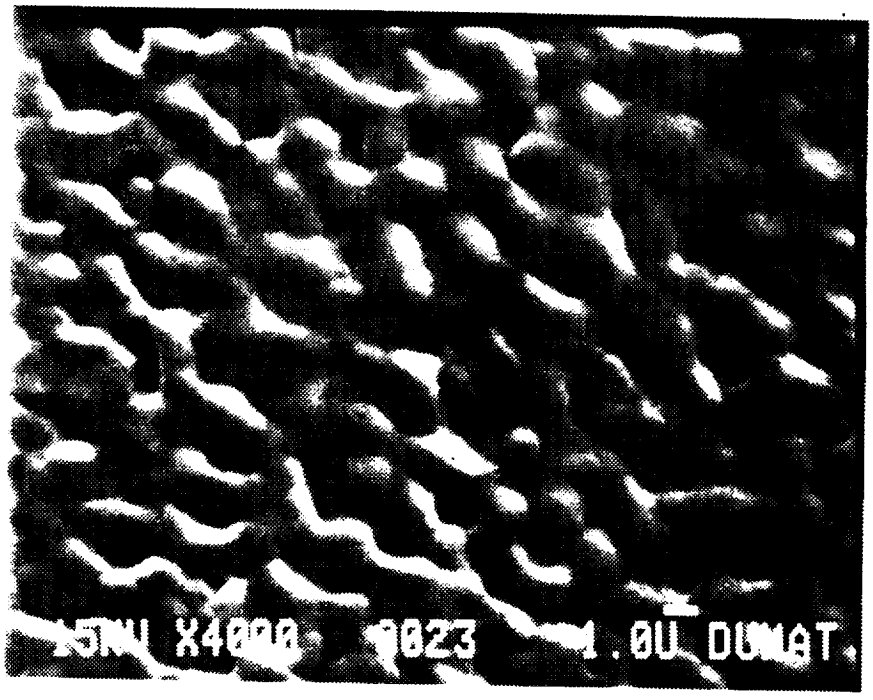


(b)

Fig 14. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature. It was coated by three layers. Then thermal treatment was performed at 600°C for 1 hour for binder removal and at 1350°C for 1 hour for sintering the alumina (Magnified to (a) 400x and (b) 4000x).



(a)



(b)

Fig 15. Treated sapphire fiber in prepared suspension having a concentration of 20% alumina at room temperature. It was coated by three layers. Then thermal treatment was performed at 600°C for 1 hour for binder removal and at 1400°C for 1 hour for sintering the alumina (Magnified to (a) 400x and (b) 4000x).